THE NICKEL BULLETIN

A MONTHLY SUMMARY OF CURRENT INFORMATION

MAY, 1958

General

	Page
Lattice Spacings and Structures of Metals	123
Measurement of Magnetostriction	123
Classification of Filler Metals Conforming to A.W.S./A.S.T.M. Specifications	124
Cermets: Bibliography of Recent Literature	124

Nickel

Nickel and Nickel Alloys: Handbook	124
Canadian Mining Industry, 1957	125
Recovery of Nickel from Alkaline Cyanide Solutions by Ion Exchange	125
Concentration of Hydrogen in Nickel under Hydrogen-Ion Bombardment	125
Solubility of Nickel in Molten Lead	125
Direct Rolling of Nickel Powder	126
Colorimetric Determination of Nickel in Ammoniacal Solutions	126
Determination of Trace Amounts of Nickel in Petroleum Fractions	127
Determination of Chromium in Nickel	127
Measurement of Corrosion by Gaseous Media: Nickel/Chlorine Reactions	127

Electrodeposition and Other Coating Methods

Plating of Nickel and Nickel Alloys	128
Corrosion-Resisting Properties of Decorative Nickel Plating	128
Atmospheric Corrosion Tests on Dull-, Semi-Bright and Bright-Nickel Coatings	129
Corrosion-Resistance of Plated Zinc-Alloy Die-Castings	130
Protective Value of Nickel/Chromium Coatings: Influence of Quality of Chromium Plating	130
Nickel Plating of Stabilizer Parts	131
Electroless Nickel as a Brazing Material	131
Cupric-Chloride Modification of the Acetic-Acid/Salt-Spray Test	131

Non-Ferrous Alloys

Nickel-Beryllium Alloys	132
Interdiffusion of Uranium and Nickel	132
Solubilities of Metals in Liquid Zinc: Literature Review	133
Aluminium-Manganese-Copper-Nickel Alloys	133
Nickel-Titanium-Carbon Alloys	134

Nickel-Iron Alloys

Solubility of Nitrogen in Iron-Nickel Alloys	134
Nickel-Aluminium-Cobalt-Iron Magnets: Development and Properties	134
Magnetic Tape produced by Powder Metallurgy	135

Constructional Steels

Inter-Relationship of Fatigue Resistance and Tensile Strength in High-Tensile Steel	135
High-Tensile Nickel-Alloy Steel in Chamber for Testing Nuclear-Powdered Aircraft Engine	136
Chemical Smoothing of Steel: Applications	136
Fracture Characteristics of Temper-Brittle Steel	136

Heat- and Corrosion-Resisting Materials

Influence of Boron and Zirconium in High-Temperature Materials	137
Effects of Forging Temperature and Heat-Treatment on Performance of Gas-Turbine Blading	137
Effect of Hot Working on Structure and Properties of High-Temperature Materials	138
Effects of Overheating on Aircraft Gas-Turbine Blading Alloys	138
Properties of Nickel-Chromium-base and Other High-Temperature Alloys	140
Titanium-Carbide-base Cermets: Engineering Properties	141
Oxidation-Resistant Coatings for Molybdenum	142
Corrosion by Vanadium Pentoxide: Influence of Titanium in High-Alloy Materials	145
Tau- and Y-Phases in Austenitic Steels	145
Effect of 'Sensitizing' Treatment on Mechanical and Physical Properties of Stainless-Steel Wire	145
Resistance of Nickel-containing Materials to Corrosion by Amine-Gas-Treating Solutions	146
Resistance of Stainless Steel to Stress Corrosion by Geothermal Steam	146
Corrosion of Metals in a Marine Tropical Atmosphere	147
„ „ Metals in Industrial Atmospheres	147
Corrosion-Resisting Chromium-Nickel-Manganese Steel	147
Corrosion by Fuming Nitric Acid: Effect of Inhibitors	148
Effect of Irradiation on Resistance of Aluminium-Nickel Alloy to Corrosion by High-Temperature Water	148
Fatigue Properties of Beryllium-Copper and Precipitation-Hardening Stainless Steels	148
Stainless-Steel and Titanium Sandwich Structures	149
'Curtain-Wall' Construction: Use of Stainless Steel	149
'Monel' Equipment for Handling Fluorine	149
Welded Stainless-Steel Tanks for Plastics-Manufacturing Plant	150
Glossary of Terms used in Reference to Corrosion	150
Determination of the Acid-Resistance of Steels by the Potentiostat Technique	150
Direct-Reading Corrosion Meter	151

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ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

GENERAL

Lattice Spacings and Structures of Metals

W. B. PEARSON: 'A Handbook of Lattice Spacings and Structures of Metals and Alloys.'

Published (1958) by *Pergamon Press*, London, New York, Paris and Los Angeles.

International Series of Monographs on Metal Physics and Physical Metallurgy, vol. 4; 1044 pp. Price £13 2s. 6d.

In recent years the study of the equilibrium structures of alloy systems has been carried out by two main methods—metallography and X-ray analysis. The results of this work have been scattered through many technical and scientific journals, and an immense effort is required to gather all the information together in a form suitable for ready reference. The Institute of Metals Bibliography on the Constitution of Alloys (*Monograph No.* 2) has been very helpful in this field, but reference to the original journals was still necessary. The Handbook now published will provide an invaluable reference when the information required is primarily that relating to the crystal structure of intermetallic phases. Although in some cases this information gives a very adequate picture of the equilibrium conditions, reference to other sources is necessary for details of the metallurgy of the alloys.

The first part of the book deals in a textual manner with the techniques of X-ray analysis, particularly the accurate measurement of lattice spacings, and explains the manner in which such measurements are used in the determination of equilibrium diagrams. A short account is given of the factors influencing the formation of intermetallic compounds and solid solutions. The various physical factors affecting lattice parameters are dealt with very fully.

The second part of the book is largely a tabulation of the results of X-ray analysis. The crystallographic data are given for structures allotted a 'Strukturbericht' type, and this is followed by tabulated lattice spacings of the elements and the intermediate phases in alloy systems.

The main part of the book gives, in alphabetical order, a summary of the X-ray work carried out on metals and on alloy systems. This review, which is quite comprehensive, includes extensive tables showing the variation of lattice parameter with composition of phases. A final section deals with similar data relating to borides, carbides, hydrides, nitrides and binary oxides.

The book will form an invaluable reference work for all engaged in X-ray analysis of metals.

Canadian Mining Industry, 1957

See abstract on p. 125.

Measurement of Magnetostriction

H. E. STAUSS: 'Method for Measuring Magnetostriction Corrected for Initial Domain Distribution and its Application to Nickel and Iron.'

Jnl. Applied Physics, 1958, vol. 29, Feb., pp. 182–4.

Measurements of magnetostriction of polycrystalline metals often show poor agreement: in the case of nickel, values in the literature vary from -25×10^{-6} to -47×10^{-6}. In general practice, linear magnetostrictive strain is determined from the change in length of the specimen after magnetization: consequently, such measurements will also include the simultaneous dimensional changes arising from other sources: e.g., from rotation of domain magnetic vectors, from a volume magnetostrictive change, from thermal expansion, and from the influence, if any, of the magnetic field on the strain-measuring equipment. Moreover, such measurements start from a poorly-defined reference state, because the unmagnetized condition is merely one in which the sum of the domain magnetic vectors is zero, and it is undefined with respect to the directional distribution of the domains.

This paper outlines a simple method of measurement which overcomes the effect of initial domain distribution.

A cubic specimen is placed directly between, and in contact with, two iron rods of similar cross-section. The composite so formed is then positioned along the common axis of two like solenoids connected in series. Strain is measured twice, with a resistance gauge, once parallel to the magnetic field, and a second time normal to the field after the specimen has been rotated through an angle of 90°. At saturation the algebraic difference is equal to 3/2 of the saturation magnetostriction. The use of this difference is claimed to eliminate or reduce a number of other experimental errors, in addition to those arising from non-randomness of initial domain distribution.

The features of the method are illustrated by a discussion of its use in determination of the saturation magnetostriction of 'A' nickel and Armco iron. Values obtained were:

nickel: $-35\times10^{-6}\pm1\times10^{-6}$
iron: $-11\times10^{-6}\pm2\times10^{-6}$

Classification of Filler Metals Conforming to A.W.S./A.S.T.M. Specifications

AMER. WELDING SOC.: 'Filler Metal Comparison Charts.'

A.W.S. Schedule A5·0–57.
Published in *Welding Jnl.*, 1958, vol. 37, Feb., pp. 128–35.

The American Welding Society receives many enquiries regarding filler metals and their classification, with particular reference to the nature of various proprietary brands, the specifications to which they broadly conform, and the sources of supply.

As a service to its members and others who use welding materials, the Society has therefore published a comprehensive group of charts showing the A.W.S./A.S.T.M. classifications and the filler metals, by brand names, which fall into the respective groups. The charts have been drawn up on the basis of information supplied by 61 makers and/or suppliers of filler materials. The American Welding Society has classified according to this information, but emphasizes that the Society assumes no responsibility for the accuracy of the listings: the manufacturer or supplier should be consulted for guarantee of compliance with specification.

Tables show A.W.S./A.S.T.M. classifications (with reputedly conforming filler materials) relating to brazing filler metals, covered stainless-steel electrodes, bare stainless-steel welding rods and electrodes, high-tensile- and low-alloy-steel electrodes, mild-steel electrodes, iron and steel welding rods, tungsten electrodes, bare aluminium welding rods and electrodes, covered nickel-alloy electrodes, covered aluminium electrodes, and copper-alloy welding rods.

Cermets: Bibliography of Recent Literature

H. C. FRIEDEMANN: 'Development of Cermets, 1950–1956.'

U.S. Atomic Energy Commission Report NYO-8527, June 7, 1957; 31 pp.

The *bibliography* was compiled to provide a background to an investigation into the slip-casting process. It is believed that use of pressureless forming, which is the aim of the slip-casting process, will be a means of avoiding, in cermets, the detrimental stresses which usually result from conventional compacting under pressure.

The scope of the bibliography, which includes 384 (unannotated) references, is sufficiently wide to make it useful in relation to any aspect of study in which cermets are involved.

'Curtain-Wall' Construction

See abstract on p. 149.

Direct-Reading Corrosion Meter

See abstract on p. 151.

NICKEL

Nickel and Nickel Alloys: Handbook

J. G. THOMPSON: 'Nickel and Its Alloys.'
Nat. Bur. Standards Circular 592, Feb. 5, 1958; 87 pp.
Superseding *Circular* 485.

This Circular, compiled on a plan similar to that used in preparation of the earlier one, constitutes a revision based on review of the literature to 1956 (with a few items from 1957), together with some information obtained from unpublished sources. Attention is directed to the fact that although the preceding *Circular* on nickel was issued in 1950, there was, at that time, a considerable amount of information which had been developed during the war and immediate post-war period, but had not yet been released for publication. Much of this material has since been made available and contributes to the revised issue.

The *Circular* is in six major sections, the scope of which is indicated below.

1. *History of Nickel*
Occurrence, extraction methods; statistics of production and consumption; available forms of nickel; metallography of nickel (including electrolytic polishing, and etching).

2. *Properties and Uses of Nickel*
Corrosion- and oxidation-resistance and other chemical properties; physical properties (including general, optical, thermal, electrical and magnetic characteristics); mechanical properties (including the influence of minor constituents); melting, casting and fabrication processes; uses of nickel, in bulk form, as plating, and as powder.

3. *Ferrous Alloys of Nickel*
Composition, properties, and some uses of low- and high-alloy steels containing nickel; cast steels and cast irons in which nickel is present; ferrous 'super-alloys' developed for high-temperature applications; nickel-iron-base alloys having special thermal-expansion properties; magnetic alloys.

4. *Non-Ferrous Alloys in which Nickel is an Essential Constituent*
Nickel-copper alloys containing more than 50 per cent. of nickel; copper-nickel types containing less than 50 per cent. of nickel; nickel-chromium alloys and nickel-chromium-iron alloys, of simple and complex types; nickel-molybdenum-base alloys; non-ferrous 'super-alloys' for high-temperature service; miscellaneous non-ferrous alloys in which nickel is present.

5. *Binary and Ternary Alloy Systems containing Nickel*
A list of *literature references* to 27 binary and 21 ternary systems of which total or partial investigations have been made.

6. *References to the Literature* (1751–1956/7)
The 800 items listed cover a selection of the major literature, arranged in date order and correlated throughout with the text of the report.

Canadian Mining Industry, 1957

'Annual Review of Canada's Mineral Industries during 1957.'

Canad. Mining Jnl., 1958, vol. 79, Feb.

Each year this journal devotes a substantial proportion of one issue to a review of the previous year's developments in Canada's mining and mineral industry. The current review, which covers the events of 1957, is in six parts, dealing, respectively, with statistics, provincial activities, principal Canadian mineral products, geological surveys, technological developments in the fields of geophysics, mining and process metallurgy. Later sections of the same issue contain brief reviews of mining progress in other parts of the world. The scope of articles of interest in relation to nickel is outlined below.

F. JOHNSON: 'Manitoba Mining Review', pp. 112–15.

Details are given of mining projects initiated, under way, or in prospect during 1957, in Manitoba Province. Information of nickel interest relates mainly to developments in the Thompson–Moak Lake area, inaugurated by The International Nickel Company, Inc. Reference is also made to the progress by Sherritt Gordon Mines and other companies.

J. C. BROWNING: 'Ontario Annual Review', pp. 115–19.

Review of the 1957 production figures and expansion programmes in Ontario. A résumé is given of the situation in relation to nickel, copper, uranium, iron, gold and cobalt, together with details of the activities of firms concerned in production of these metals.

R. J. JONES: 'Nickel in Canada—1957', pp. 131–2.

Review of Canadian nickel-production figures for 1957 and of the development plans of individual nickel-producing firms. Future trends are discussed, and consideration is given to the effects, on the Canadian nickel industry, of a nickel output which would be surplus to world demand.

Lattice Spacings and Structures of Metals

See abstract on p. 123.

Recovery of Nickel from Alkaline Cyanide Solutions by Ion Exchange

J. AVESTON, D. A. EVEREST, N. F. KEMBER and R. A. WELLS: 'Recovery of Gold, Silver and Nickel from Alkaline Cyanide Solutions by Means of Weak-base Ion-Exchange Resins.'

Jnl. Applied Chemistry, 1958, vol. 8, Feb., pp. 77–86.

Gold as cyanoaurate is selectively adsorbed by weak-base resins from alkaline cyanide liquors containing base-metal cyanides, and can be quantitatively eluted with aqueous sodium thiocyanate. Interference from sulphur-containing anions, particularly thiocyanate, which are present in gold-ore leach liquors, may be overcome by incorporation of 6–10 per cent. of strong-base groups into the weak-base resin. Resins containing about equal numbers of strong- and weak-base groups are reasonably selective for nickel and silver, which are normally adsorbed together. They may be eluted with either sodium thiocyanate or sodium nitrate, made alkaline with ammonia or sodium cyanide. A possible method for the large-scale recovery of gold, nickel and silver from pregnant cyanide liquors is put forward.

Concentration of Hydrogen in Nickel under Hydrogen-Ion Bombardment

J. MORRISON and J. J. LANDER: 'The Concentration of Hydrogen in Nickel under Hydrogen-Ion Bombardment.'

Jnl. Electrochemical Soc., 1958, vol. 105, Mar., pp. 145–51.

Previous investigations had shown that very high concentrations of hydrogen were produced in zinc oxide by means of hydrogen-ion bombardment. This phenomenon was ascribed to (*a*) the efficient penetration, by ions of sufficient velocity, of a surface barrier which inhibits normal reaction, and (*b*) the efficacy of the surface barrier in preventing escape of dissolved hydrogen. This report presents the results of an extension of the investigation to nickel specimens, the surfaces of which were (*a*) 'clean', (*b*) oxidized, or (*c*) coated with a film of barium oxide. The concentration of hydrogen dissolved in the three conditions was measured, using a nickel diffusion tube as the cathode in a glow discharge.

The conclusions drawn from the results are summarized below:

No increase above the normal concentration was observed when 'clean' nickel was bombarded. These results indicate that there is no severe barrier to the escape of hydrogen from a 'clean' nickel surface.

When the nickel was coated with nickel oxide or barium oxide increases of several orders of magnitude were observed in the range near 200°C. It is postulated that in this case also energetic ions penetrate the outer surfaces of these materials efficiently but are then trapped in the interior by surface barriers. The data indicate that if the metal surfaces are contaminated it is not possible to apply normal values of concentration to nickel (and probably to many other metals) when operating under hydrogen-ion bombardment.

Measurement of Magnetostriction: Application to Nickel

See abstract on p. 123.

Solubility of Nickel in Molten Lead

T. ALDEN, D. A. STEVENSON and J. WULFF: 'Solubility of Nickel and Chromium in Molten Lead.'

Trans. Met. Soc., Amer. Inst. Mining Engineers, 1958, vol. 212, Feb., pp. 15–17; *T.P.* 4593*E*.

Increased use of molten metals of low melting point as heat-transfer media for coating, joining and other purposes has awakened fresh interest in the solubility of structural metals in such liquids. Study of the literature indicated that there have been few accurate determinations of liquidus curves at the low-melting end of the relevant binary diagrams,

particularly those in which lead is a constituent. This finding promoted the investigation reported in the present paper, which covers determination of the solubility of nickel, chromium, tungsten and molybdenum in molten lead. *Inter alia*, the liquidus curve for nickel established by PELZEL (*Metall*, 1955, vol. 9, Aug., pp. 692–4; *Nickel Bulletin*, 1956, vol. 29, Nos. 1–2, p. 7) was checked and extended to 1244°C.

Use was made of a sampling technique which enabled numerous points on a curve to be obtained from a single furnace run: the composition of the liquid samples taken was determined by chemical analysis. Data derived for the solubility of nickel and of chromium in lead are shown in tabular and graphical form. No data are recorded for tungsten or molybdenum, because both these elements exhibited very low solubility (less than 0·005 wt. per cent. at 1200°C.). The solubility of nickel in lead was found to be 8·03 per cent. at 1244°C. and 0·15 per cent. at 372°C. The differential heat of solution, calculated from these data, was 2110 cal. per mol.

In the case of chromium 0·1 per cent. was soluble in lead at 908°C. and 0·16 per cent. at 1210°C.

Solubility of Nickel in Liquid Zinc

See abstract on p. 133.

Interdiffusion of Uranium and Nickel

See abstract on p. 132.

Resistance of Nickel to Corrosion by Amine-Gas-Treating Solutions

See abstract on p. 146.

Direct Rolling of Nickel Powder

The following abstract is published of a paper presented at The Institute of Metals Division of The Metallurgical Society of The American Institute of Mining Engineers.

J. A. LUND: 'Roll Compacting of Pure Nickel Strip from Powder.'
Jnl. of Metals, 1958, vol. 10, Feb., p. 95.

'Nickel strip of excellent mechanical properties has been prepared by the direct roll-compacting of pure nickel powder, followed by sintering and cold re-rolling of the as-compacted product.

'Coherent green strip was produced in the initial roll-compacting operation, and the effects of metal-powder particle size, roll gap, and rolling speed, on the density of the as-compacted strip, are reported. Green-strip density varied from 77 per cent. to 88 per cent. of the theoretical density of pure nickel. The microstructure of the green strip indicated that the hard nickel-powder particles had not been plastically deformed in the rolls, but that many particles had fragmented in a brittle manner. The coherency of the as-compacted material, which permitted it to be handled quite readily in short lengths, has been attributed mainly to mechanical interlocking of the metal particles.

'The effects, on strip properties, of sintering at several temperatures for various times, and of progressive cold re-rolling after sintering, were studied. Sintering temperatures of 1900° to 2250°F. (1038° to 1232°C.) for times of 30–15 minutes were found to be suitable, followed by a total cold-reduction of 65–75 per cent. in the thickness of the sintered strip, with intermediate and final annealing at 1600°F. (870°C.) for 30 minutes. With this treatment, strip has been obtained with values of density, tensile strength and ductility at least as high as those reported for cast, rolled and annealed pure nickel. Best properties were observed in strip which had been roll-compacted originally from the finer grades of nickel powder. Other roll-compacting variables appeared to have almost no effect on the properties obtainable after sintering and extensive cold re-rolling.

'Densified and annealed strip has been reduced up to 45 per cent. in thickness by cold-rolling without annealing. Tensile strength of the strip rose to 85,000 p.s.i. (38 t.s.i.; 60 kg./mm.^{2}) with this treatment.'

If and when this paper is published in full, notification will be made in *The Nickel Bulletin*.

Nickel for Handling Fluorine

See abstract on p. 149.

Colorimetric Determination of Nickel in Ammoniacal Solutions

D. A. SKOOG, M.-G. LAI and A. FURST: 'Quinoxaline-2,3-Dithiol as a Colorimetric Reagent. Determination of Nickel in Ammoniacal Solutions.'
Analytical Chemistry, 1958, vol. 30, Mar., pp. 365–8.

The authors describe a method which, it is suggested, may have some advantages over procedures in which dimethylglyoxime is employed.

For introductory information on the preparation of the reagent proposed, reference is directed to work by MORRISON and FURST (*Organic Chemistry*, 1956, vol. 21, p. 470). These investigators found that quinoxaline-2,3-dithiol formed chelates with a number of the transition elements, and that with ammoniacal solutions of nickel a pink colour is produced which is sensitive to low concentrations of the metal.

Further work showed that this compound constitutes a satisfactory colorimetric reagent for detection and quantitative determination of nickel. It is found that concentrations as low as 0·03 p.p.m. can be detected in aqueous ammoniacal solution and that nickel contents in the range 0·1–3 p.p.m. can be estimated with a relative accuracy of 1 per cent. The method has the added advantage that the colour produced is stable for several hours and that solutions of the reagent, correctly prepared, remain stable for several days.

Full details of procedure are given, together with discussion of the effects of several variables (ammonia concentration, concentration of the reagent, age of the reagent solution). Interference is caused by the presence of silver, copper, cobalt and manganese, but not by a considerable number of other ions. Degree of interference (or immunity from it) is demonstrated for some twenty foreign ions.

Determination of Trace Amounts of Nickel in Petroleum Fractions

J. HANSEN and C. R. HODGKINS: 'Wet-Ash Spectrochemical Method for Determination of Trace Metals in Petroleum Fractions.'

Analytical Chemistry, 1958, vol. 30, Mar., pp. 368–72.

A well-recognized problem in the petroleum industry is the interference in operation of catalytic processes resulting from degradation of the catalyst by poisoning. The poisons responsible are in many cases introduced with the feed stocks: for example, even the minute traces of nickel, vanadium and iron in the feed can cause catalyst degradation to an economically important degree.

Development of inhibitory methods must depend closely on techniques for the highly accurate estimation of the small amounts of metal present. For this purpose spectrochemical analysis of wet-ash residues has an advantage over colorimetric methods because of its greater sensitivity, and because, with the spectrochemical method, it is possible simultaneously to determine several elements. In addition, the need for a calculated reagent blank correction is eliminated.

This paper reviews some work which has been done in selection of materials suitable for use as a matrix on which to deposit the trace metals, a technique by which the metals of the sample become, in effect, impurities in the matrix material. Requirements which must be satisfied by the matrix material are briefly reviewed, and a summary is made of experience reported by other investigators with regard to various media which have been tried for that purpose. A detailed description is then given of procedure developed by Esso Research and Engineering Company, by which copper-metal powder is employed as a carrier matrix, on which nickel, vanadium and iron trace contaminants of petroleum fractions are deposited when the sample is decomposed by a sulphuric-acid, wet-ash method. By this procedure it is possible to determine as little as 0·05 p.p.m. of these elements in a 10-g. sample. It is noted that the addition of spectroscopically pure graphite powder to the copper-residue mixture reduces sputtering during excitation and results in more reproducible spectrograms. Full details of procedure are given, together with illustrations of equipment used, and reports of check analyses. It is stated that determination of the three elements (nickel, vanadium and iron) requires between $\frac{3}{4}$ and $1\frac{1}{2}$ man-hours when 20–25 samples are run as a batch.

Determination of Chromium in Nickel

C. L. LUKE: 'Photometric Determination of Chromium in Electronic Nickel.'

Analytical Chemistry, 1958, vol. 30, Mar., pp. 359–61.

The following experience is recorded with regard to attempts to develop a method suitable for estimation of the small amounts of chromium liable to occur in nickel used for electronic components.

In view of the low percentages of chromium involved, the presumption was that a photometric technique would be most suitable. Reporting preliminary experiments leading up to the development of the recommended method, the following observations are made:

'Unsuccessful attempts were made to determine the chromium directly in acid solution without prior separation of the nickel or any of the impurities therein, except silicon or tungsten. The photometric dichromate method proved to be too insensitive. The diphenylcarbazide method was found to be sufficiently sensitive, but copper caused low results, and no suitable means was found of destroying the permanganate formed during the oxidation of chromium. Some reduction of the dichromate invariably occurred.

'Methods for isolating the chromium were next considered. An attempt was made to separate it from the bulk of the nickel, cobalt, copper, manganese and magnesium by precipitating it from ammoniacal solution as chromic hydroxide, using 10 mg. of ferric iron as a co-precipitant. Recovery of the chromium was incomplete.'

Eventually a successful method of separation was evolved in which copper is removed by dithizone-chloroform extraction, chromium is oxidized to the sexivalent state, and nickel, cobalt, iron, titanium, and aluminium are removed by an ammonium-hydroxide separation, the nickel and cobalt being separated in the form of their difficultly-soluble ammonium-perchlorate salts. The chromate in the filtrate from this separation is then determined by the diphenylcarbazide method.

This method is applicable for estimation of chromium over the range 0·001 to 0·02 per cent. Confirmatory check tests on synthetic nickel specimens of known chromium content and on Bureau of Standards samples are reported. Among the features specially discussed by the authors are the critical nature of the step in which chromium is oxidized to the sexivalent state, and the importance of avoiding interference by copper, by removing it while the chromium is still in the trivalent state.

Measurement of Corrosion by Gaseous Media: Nickel/Chlorine Reactions

J. D. McKINLEY and K. E. SHULER: 'Kinetics of the Reaction between Nickel and Chlorine above 1100°K. (827°C.).'

Proc. XVIth Congress of Pure and Applied Chemistry, Paris, 1957.

Abstract in *Corrosion*, 1958, vol. 14, Feb., p. 95.

The authors describe a technique for measurement of the rate at which a metal surface is attacked by a corrosive gas, and demonstrate its application to the investigation of reactions occurring between nickel and chlorine at elevated temperatures. The procedure used, which has been developed at the National Bureau of Standards, employs time-lapse photography of the reacting solid, to give permanen records of the progress of the reaction at given intervals. Study of nickel/chlorine reactions, over

the range 1100°–1700°K. (827°–1427°C.) showed that the rate of change is linear with time. The rate of the reaction is proportional to the chlorine pressure and is extremely rapid.

ELECTRODEPOSITION AND OTHER COATING METHODS

Plating of Nickel and Nickel Alloys

W. W. SELLERS and C. B. SANBORN: 'Electroplating on Nickel and Nickel Alloys.'

Reprint of paper from 44*th Ann. Tech. Proceedings, Amer. Electroplaters' Soc.*, 1957, pp. 36–41. Issued by INTERNATIONAL NICKEL CO., INC.

See abstract in *Nickel Bulletin*, 1958, vol. 31, No. 4, pp. 101–5.

Corrosion-Resisting Properties of Decorative Nickel Plating

C. H. SAMPLE: 'Developments in Decorative Plating.' *Soc. Automotive Engineers, Preprint* 220, Jan. 1958; 14 pp.

This paper arose out of complaints of the quality and appearance of decorative plating on motor cars.

The paper falls into two sections. The first consists of a short historical survey, covering discussion of the significance, in the evolution of modern decorative electroplating techniques, of such fundamental advances as the development of the Watts nickel solution, bright chromium plating, and bright and semi-bright proprietary nickel-plating solutions. Reference is made also to improvements effected by the introduction of more efficient techniques for preparation of the basis metal, the inauguration of specifications for plated coatings, improvement of test methods, and the introduction of automatic cleaning and plating machines.

The second part of the paper directs attention to data (some previously unpublished) which have been derived from atmospheric corrosion tests on decorative nickel/chromium coatings, carried out under the aegis of the American Society for Testing Materials, The International Nickel Company, Inc. and other organizations. These tests, in which, *inter alia*, comparison was made of the performance of copper/nickel/chromium coatings and of nickel/chromium coatings, confirmed conclusively that the principal factor determining the corrosion-resistance of these composite plating systems is the thickness of the nickel underlayer. Moreover, in each of the thicknesses of nickel coating tested (1·0, 1·5 and 2·0 mils: 0·025, 0·037 and 0·05 mm.) deposition of the nickel in two stages (the first layer buffed, or annealed and buffed) markedly increased resistance to corrosion by marine or rural atmospheres. Little or no improvement was, however, observed in the behaviour of double-plated specimens exposed to industrial atmospheres.

This variation in performance is explained in terms of the more severely corrosive conditions prevailing in industrial atmospheres, in which failure can result not only from porosity present in the coatings, but also as a result of the pinholes created by concentrated localized corrosion. On this basis, the superior performance of the double-layer nickel deposits in rural or marine atmospheres can be ascribed to the relative absence of residual porosity. (In connexion with these experiments, it is stated that a schedule of recommended practice for the production of double-layer coatings is to be prepared by the American Society for Testing Materials.)

The author emphasizes that the conclusions apply only to buffed, dull-nickel deposits and that the results of subsequent test programmes (in particular the current programme of the American Society for Testing Materials Committee in which both bright- and buffed dull-nickel undercoatings are being evaluated) indicate that the corrosion-resistance of bright coatings is variable, and generally inferior to that of a buffed dull-nickel undercoating of the same thickness. The protective value of composite coatings has again been found to increase with increase in thickness of the nickel deposit, though the protection conferred by the thickest bright-nickel coating (2·5 mils: 0·032 mm.) is less than would be anticipated from comparable dull-nickel deposits. The results suggest that minimum coating thicknesses of the present A.S.T.M./A.E.S. specifications (which reflect the performance of buffed dull nickel) may be inadequate when applied to bright-nickel deposits.

Finally, the author considers the influence, on the durability of bright-nickel/chromium deposits, of the final chromium-plating operation. The results of tests in which thickness of chromium was included as a variable showed that a six-fold increase in the normal thickness of the final deposit (i.e., from 0·01 to 0·06 mils: 0·00025 to 0·0015 mm.), when applied over 1·5 mils (0·037 mm.) of bright nickel, resulted in a marked improvement in corrosion-resistance. In explanation of the deleterious effect of chromium plating when deposited over bright nickel, the author advances a theory linked with the phenomenon of hydrogen embrittlement, i.e., that a hard bright-nickel coating, perhaps containing unduly high internal stresses, might be susceptible to spontaneous hydrogen cracking when chromium plated. In support of such an explanation, he cites the enhanced protection achieved by increasing the thickness of the chromium deposit: it would be reasonable to expect that any cracks, occurring during the first few seconds of chromium plating would be filled by the stage at which some 0·06 mils: (0·0015 mm.) of chromium had been deposited.

Although the paper is to some extent a criticism of present-day bright-nickel plating, it is emphasized

that bright-nickel plating plays an indispensable rôle in the field of decorative coatings, and that, from the economic aspect alone, the industry would be most unlikely to revert to buffed dull-nickel deposits as a basis for final chromium plating.

Atmospheric Corrosion Tests on Dull-, Semi-Bright- and Bright-Nickel Coatings

J. EDWARDS: 'Atmospheric Exposure Tests of Different Types of Electrodeposited Nickel.'

Product Finishing, 1958, vol. 11, Mar., pp. 58-66.

This paper may conveniently be read in association with the review by SAMPLE referred to in the preceding abstract. The economic benefits derived from bright-nickel plating are well known to be substantial, but the paper by Sample draws attention to some doubts which exist with respect to the service durability of bright and semi-bright coatings, and refers to the superior corrosion-resistance which dull-nickel deposits are reported to have shown when used as underlay to chromium in atmospheric tests. Reference to the literature has not, however, clearly established that the two types of nickel deposit show significant differences in this respect.

The work described in the present paper, carried out by British Non-Ferrous Metals Research Association, was therefore initiated with the sole aim of comparing the resistance to atmospheric corrosion conferred on steel pressings by various types of nickel coating, deposited under a final layer of chromium of normal thickness.

Seven types of nickel coating were investigated: *dull nickel* (produced from a Watts bath), *organic bright nickel* (produced from the proprietary solutions 'Super Gleamax', 'Efco-Udylite No. 31', 'Silvercrown' and 'Perglow'), *organic semi-bright nickel* (produced from the appropriate 'Perglow' solution), and *cobalt-nickel* (produced from a 'Lumax' solution). In addition, atmospheric corrosion tests were made on nickel deposits of all these grades, in the form of foils detached from the basis metal and carrying no top coating of chromium. For purposes of comparison, specimens of rolled nickel sheet were also tested. Freshly prepared solutions were used to plate each batch of specimens, and the plating conditions (full details of which are given) were regulated to give deposits 0·0012 in. (0·03 mm.) thick.

The specimens were exposed, for a period of one year, at sites representative of industrial and marine atmospheres, one in London, the other at a coastal site in the south of England. Exposure and inspection procedures are described. Assessment of the type and extent of corrosion was made in three ways: (1) deterioration in decorative value was estimated by the appearance of the specimen in relation to duration of exposure; (2) a detailed quantitative assessment of corrosion-resistance was made by recording the size and number of points of attack (this method was suitable for use only in the first few months, after which the pits formed were too small and numerous to be effectively recorded); (3) loss in weight was measured.

Results are presented in the form of tables listing percentage ratings calculated from points allotted on the general (decorative) assessment, and on average loss in weight, in both cases after one year's exposure. Degree and extent of corrosion are discussed. The conclusions drawn are summarized below.

The results reported indicate, that, within the scope of the test conditions, the resistance to corrosion, in industrial and marine environments, of nickel deposited from sulphate-chloride solutions is virtually independent of the additions made to promote brightening, levelling, low stress, etc. This conclusion is valid whether or not the deposit has an outer coating of chromium, provided that the quality of the chromium on the different types of nickel undercoat is the same. It is, however, known that conditions of deposition and thickness of the chromium deposit have a marked effect on the corrosion-resistance of composite coatings. For example, plating at a slightly higher temperature (about 55°C.) in a normal chromic-acid/sulphuric-acid solution, requiring a current density of about 30 amp./sq. ft. (3·24 amp./dm.^{2}), permits deposition of thicker bright deposits free from cracks. It is possible, by various methods, to obtain much thicker crack-free deposits, which can substantially reduce the severity of corrosion of the nickel undercoat, but such coatings are usually dull and require polishing. It is considered reasonable to assume that the type of nickel used under such thicker deposits would have no more effect on corrosion-resistance than was observed in the work reported, in which conventional thin chromium outer coatings were used.

Unsuitable conditions of nickel deposition or the presence of impurities in the nickel-plating solutions could give rise to defects leading to premature failure, but such defects were avoided in preparing the specimens used in these tests. It is usually possible in practice to maintain clean plating conditions, but the question arises whether, with increasing length of service of the solution, the apparently inevitable accumulation of impurities in nickel solutions containing organic addition agents leads to deterioration in the durability of deposits. (A paper by the same author, dealing with service life of bright-nickel-plating solutions, was recently presented before the Institute of Metal Finishing. It will be abstracted in the June issue of *The Nickel Bulletin*.)

Failure of all the deposits examined in this series of tests appears to have taken place by penetration of the chromium film, followed by local corrosion of the nickel deposit. Penetration of the nickel coating was at first relatively rapid while there were only a few pits, but became much slower when the number of pits became greater. There is no evidence that pre-existing porosity of the nickel layer played any important part in the breakdown mechanism, a finding which is consistent with the experimental data reported and with views expressed by WESLEY and KNAPP (*Trans. Inst. Metal Finishing*, 1954, vol. 31, p. 267).

Corrosion-Resistance of Plated Zinc-Alloy Die-Castings

V. E. CARTER and J. EDWARDS: 'Service Blistering of Plated Zinc-Alloy Die-Castings.'

Metal Industry, 1958, vol. 92, Mar. 7, pp. 191-2, 194.

Service blistering of nickel/chromium-plated die-castings is generally believed to arise from corrosion of the basis metal, following penetration of the coating. It is, however, sometimes suggested that blisters may occur in service without penetration of the coating having taken place. This paper reports tests, carried out by the British Non-Ferrous Metals Research Association, to determine more closely the cause, nature and extent of blisters occurring on a representative series of zinc-base die-castings plated with various thicknesses of copper + nickel (dull or bright) + chromium.

The specimens were exposed for 8 months in an industrial atmosphere, being examined at monthly intervals. Their behaviour is rated in six gradings, ranging from 'complete freedom from blistering' to 'complete failure'. Ratings of corrosion for the 8 months are correlated with the thickness of the copper and nickel coatings, and an estimate is made of the probable behaviour over a period of one year.

It is shown that resistance to blistering is strongly dependent on the thicknesses of the nickel deposit. and that it is probably little affected by variations in the thickness of the copper underlayer. The curves indicate that durability of the coating increases rapidly as the thickness of nickel is increased, especially up to about 0·0009 in. (0·023 mm.). These figures suggest that at least 0·0009 in. of nickel is required on zinc-alloy die-castings and, in view of the estimated figures for corrosion over a year period, it is probable that a rather greater thickness of nickel (possibly 0·001 in.: 0·025 mm.) should be specified for outdoor use.

With regard to the origin of the blisters, the following observations were made:

'The first blisters to appear on every plated casting exposed in the tests were carefully examined under the microscope. A small hole in the coating was visible at the top of every blister. In some cases the initial pit had been enlarged by cracking of the coating caused by the eruption of the blister, and white corrosion product could be seen in the hole. Surface examination of some small blisters failed to show conclusively that the pit penetrated the coating, but cutting open such blisters revealed that they were full of corrosion product, indicating that there must have been a perforation in the coating, permitting the escape of gaseous products of corrosion (hydrogen) and probably providing the means of entry for corrosive agents.

'Process blisters, occurring during or shortly after plating, usually contain very little solid corrosion product, and since they are formed in most cases by the pressure of gas generated (or expanded, at least) beneath the coating, the coating must not be perforated. The fact that the blisters examined in the present work were perforated, and were full of solid corrosion product, demonstrated conclusively that they were formed by corrosion of the basis metal following penetration of the coating. Since not one blister was found without these characteristics, it seems most unlikely that blisters of any other kind (such as delayed process blisters) contribute appreciably to the deterioration of plated zinc-alloy die-castings in service.'

Nickel and Nickel Alloys: Handbook

See abstract on p. 124.

Protective Value of Nickel/Chromium Coatings: Influence of Quality of Chromium Plating

H. BROWN, M. WEINBERG and R. J. CLAUSS: 'Corrosion Studies with Nickel/Chromium Plate.'

Plating, 1958, vol. 45, Feb., pp. 144–50.

Among the factors influencing the durability of electrodeposited nickel/chromium or copper/nickel/chromium coatings, the significance of the thickness and quality of the nickel undercoat is well recognized. This paper emphasizes the complementary importance of the quality and thickness of the final chromium coating.

The paper opens with a discussion of the nature of the corrosion cell set up when the corrodents in industrial atmospheres penetrate through cracks in the chromium outer coating and reach the nickel underlay. In these conditions the surface of the chromium plating becomes the cathode, and the underlying nickel exposed in the cracks or pores of the chromium becomes a series of tiny anodes. It is once more demonstrated that the thicker the nickel coating, or the more resistant the type of nickel can be made to anodic attack, the better is the protection provided for the underlying basis metal.

The authors report tests indicating the importance of ensuring deposition of the chromium in conditions, and to a thickness, which will minimize stress cracking and porosity; comparison is also made of the degree of resistance to anodic corrosion of various types of nickel underlay. The data on which the recommendations are based were obtained by outdoor exposure, in an industrial atmosphere, of chromium-plated nickel foils, of nickel/chromium-plated steel, copper/nickel/chromium-plated steel and plated zinc-base die castings. The results of the weathering tests were correlated with behaviour under accelerated test conditions, using the acetic-acid/salt-spray method, 'Corrodkote' (a kaolin coating containing ferric chloride, ammonium chloride and cupric chloride), and nitric-acid vapour.

Earlier investigations have shown that the pore pattern of thin chromium coatings, and the stress-cracking pattern of thicker deposits, varies markedly with the temperature of the solution from which the chromium is deposited, with the ratio of chromic acid to sulphate, and with other factors such as the nature and structure of the metal over which the chromium is deposited and the degree of the stress present. Results obtained with chromium coatings in the corrosion tests reported confirm that bright plating deposited from electrolytes held at about

131°F. (55°C.) shows minimum porosity and stress cracking at thicknesses of 0·02–0·08 mils (a preferred range is 0·05–0·08 mils). Further, the tests indicated that deposits made from solutions containing high ratios of chromic acid to sulphate, e.g., 125–150 : 1, which had been shown to exhibit minimum porosity for a given thickness of coating, afforded the best protection in the corrosive conditions used in the present investigation.

Some of the results relating to comparison of various types of nickel underlay are to be published later, but the authors report in this paper a comparative study of chromium coatings deposited over various types of sulphur-containing and sulphur-free nickel undercoats. In all cases the sulphur-free underlays gave the better protection.

The contrast between the protection obtained with bright chromium plating deposited under varying conditions is further demonstrated by illustrations of corrosion pits in nickel/chromium and copper/nickel/chromium coatings on steel and on zinc-base die-castings.

Oxidation-Resistant Coatings for Molybdenum

See abstract on p. 142.

Nickel Plating of Stabilizer Parts

'Stabilizing Small Ships.'

Engineering, 1958, vol. 185, Jan. 24, p. 122.

Although activated-fin stabilization of large ships is now an accepted feature of shipbuilding practice, the technique has not been generally adopted in ships below 1,000-tons' displacement, due to the difficulty of installing the equipment in the restricted space available. Considerable interest has therefore been aroused by the development (by Saunders-Roe (Anglesey), Ltd., in co-operation with William Denny Brothers and Co. and Brown Brothers and Co., Ltd.) of a range of units adaptable to a wide variety of small ships and boats. This article contains brief notes on the main features of a typical unit, with diagram.

The stabilizers are designed to give long life with the minimum of maintenance: suitable protection against corrosion and wear has been provided where necessary. The drive shaft, which is in 3 per cent. chromium-molybdenum steel, is, for example, nickel-plated by the 'Fescol' process.

Electroless Nickel as a Brazing Material

P. PATRIARCA, G. M. SLAUGHTER and W. D. MANLY: 'Electroless-Plated Brazing Alloys.'

Oak Ridge Nat. Laboratory Report 2243, Mar. 19, 1957; 12 pp.

Consideration of the composition and constitution of the nickel-phosphorus alloy deposited by the electroless plating process indicated its potential usefulness as a high-temperature brazing alloy. In the exploratory experiments described in this report a series of nickel-chromium-molybdenum stainless-steel strips were plated with a nominal thickness of 0·001 in. (0·025 mm.), using the Brenner technique. Photomicrographs of the deposited coating (illustrated in the report) demonstrate its density. The hardness of the coating was Vickers 361 and it was brittle. Brazing experiments with the plated strip demonstrated the possibility of obtaining sound joints by this means and tests of the brazed joints for 500 hours in air at 1500°F. (815°C.) showed the joints to be satisfactorily resistant to oxidation. Static tests in sodium, for 100 hours at the same temperature, indicated, however, that brazed joints so produced would not be suitable for use in that type of environment.

Attempts to improve the corrosion-resistance of the nickel-phosphorus brazing alloy were made by superimposing, prior to brazing, an overlay of 0·0002 in. (0·005 mm.) of chromium. Joints made with 'Inconel' strip so treated indicated benefit from incorporation of chromium: they showed good resistance to both air and sodium at 1500°F. (815°C.).

The general conclusion is that deposits made by the electroless plating process exhibit excellent wettability and flowability in conjunction with such high-temperature materials as austenitic stainless steels or 'Inconel', when heated above the eutectic temperature in dry hydrogen.

Chemical Smoothing of Steel before Electroplating

See abstract on p. 136.

Cupric-Chloride Modification of the Acetic-Acid/Salt-Spray Test

G. L. SYKES: 'Accelerated Salt-Spray Testing of Plated Parts.'

Automotive Industries, 1957, vol. 117, Dec. 1, pp. 54–5, 113.

Tests for rapid evaluation of the corrosion-resistance of electrodeposited coatings have in recent years been the subject of much research. A variety of tests have been evolved and interest has at various times centred on the 5 per cent. and 20 per cent. (neutral) salt-spray tests, the sulphur-dioxide test, and the acetic-acid/salt-spray test. Critical evaluation of these and other tests has recently appeared in papers by HOOPER (*Electroplating and Metal Finishing*, 1957, vol. 10, Dec., pp. 403–8; *Nickel Bulletin*, 1958, vol. 31, No. 2, p. 44) and by EDWARDS (*Trans. Inst. Metal Finishing*, 1958, vol. 35; *Nickel Bulletin*, 1958, vol. 31, No. 3, pp. 73–4). In view of the wide range of corrosive environments to which plated parts on cars are subjected in service, particularly intensive efforts have been made in the motor-car industry to develop an accelerated corrosion test capable of yielding results which will permit accurate forecast of service behaviour. No method so far devised has proved fully satisfactory, but this paper gives details of a test which, in the author's opinion, holds promise of simulating service conditions.

After briefly reviewing the requisites of an accelerated

corrosion test, an account is given of the factors which led up to the development of a modified test by the Cadillac Motor Car Division of General Motors Corporation. With the introduction of the 1956 model, the neutral salt-spray test was replaced by the acetic-acid/salt-spray test as a means of evaluating corrosion-resistance of plated parts, and as a result of this move, defects in the copper/nickel/chromium deposits or anodized parts were more effectively revealed and quality was gradually improved. It soon became apparent, however, that these advantages were outweighed by one overwhelming limitation of the acetic-acid/salt-spray test: the need for examining and rating the samples every 24 hours, for 8 consecutive days. Remedial action was hampered, and the number of specimens capable of being tested was limited, by the long duration of the test. A series of investigations was therefore initiated with the aim of devising a modified test which would, in a 24-hour period, produce results comparable to those achieved in 192 hours by means of the acetic-acid/salt-spray test.

Preliminary experiments indicated that action of the salt-spray test was likely to be accelerated by (1) addition of ferric chloride and/or cupric chloride to the solution, and (2) raising the temperature in the fog chamber. Ferric chloride proved unusable on account of precipitation of iron; reduction of the pH value of the solution to below 3·2 produced an etching effect rather than the red-rust type of failure characteristic of service corrosion of copper/nickel/chromium deposits. The conditions finally regarded as optimum for the modified test are given below:

Saturator-Tower Temperature: 130°-135°F. (55°-57°C.)
Salt-Fog-Chamber Temperature: 120°F. approx. (50°C.)
Fog-Collection Rate: 1-2 ml./hr./80 sq. cm.

Composition of Solution:

Distilled water	5 U.S. gallons (19 litres)
Sodium chloride	5 wt. per cent.
Cupric chloride	5·5 g. $CuCl_2.2H_2O$
Iron	less than 0·0001 per cent.

pH 3·2
(adjusted with glacial acetic acid)

In 24–48 hours this modified test produced corrosion on anodized aluminium and copper/nickel/chromium-plated steel, with results similar to those achieved by means of the acetic-acid/salt-spray test only after 192 hours of testing. Results of other tests on plated aluminium, zinc and steel are also presented. Tabular data illustrate the rating method used and suggestions are put forward as a basis for establishment of acceptance standards for various thicknesses of deposits. Experience to date indicates that the test goes far to satisfy essential requirements. It has been recently accepted as standard by General Motors Corporation, but it is acknowledged that there is still room for development in such features as rating procedure, establishment of acceptance standards and correlation of field experience with experimental results.

NON-FERROUS ALLOYS

Nickel and Nickel Alloys: Handbook

See abstract on p. 124.

Nickel-Beryllium Alloys

The following abstract is published of a paper presented at the Institute of Metals Division of the Metallurgical Society of The American Institute of Mining Engineers.

S. H. GELLES, R. E. OGILVIE and R. A. KAUFMANN: 'Solid Solubilities of Iron and Nickel in Beryllium.' *Jnl. of Metals*, 1958, vol. 10, Feb., p. 99.

'The solid-solubility limits of iron in beryllium were determined between 850° and 1200°C., by analysis of differential-type multi-phase diffusion couples, using an X-ray absorption technique. The maximum value of the solubility limit was found to be $0·92 \pm 0·02$ at. per cent. (5·46 wt. per cent.) at the eutectic temperature, 1225°C. The solubilities of nickel in beryllium were determined between 900° and 1200°C. by the same technique, and the maximum solubility was found to be $4·93 \pm 0·01$ at. per cent. (25·2 wt. per cent.) at the eutectoid temperature, 1065°C.

'A high-temperature phase, which decomposes eutectoidally at 1065°C., was found to exist in the beryllium-nickel system at a composition of approximately 8 at. per cent. nickel (36 wt. per cent.) by diffusion-couple analysis. No mention of this phase has been made in previous papers on the beryllium-nickel system, although its presence has been confirmed in this work by thermal analysis and metallographic analysis of the structure resulting from the eutectoid decomposition.'

If and when this paper is published in full, notification will be made in *The Nickel Bulletin*.

Solubility of Nickel in Molten Lead

See abstract on p. 125.

Interdiffusion of Uranium and Nickel

S. AAS and A. F. STEINEGGER: 'Interdiffusion of Uranium and Nickel.'

Tidsskr. Kjemi, Bergvesen og Metallurgi, 1957, vol. 17, No. 10, pp. 171–4.

In some cases in which aluminium has been used as the canning material in a metallic-uranium fuel element, reaction between the two metals at higher temperatures has caused difficulties in reactor operation and it has been necessary to introduce a diffusion barrier between the canning material and the fuel. For many applications, however, a metallurgical bond between the canning material and the fuel is desirable and it would be advantageous if the diffusion barrier were of such a nature that it would also serve as a bonding medium. Nickel has been proposed for such use, and the investigation reported in this paper was carried out as part of a programme designed to determine the feasibility of using this metal. The

aim of the investigation was thus of a practical nature, for which it was considered sufficient to determine the increase in width of the total inter-diffusion zone as a function of time and temperature, and the relative diffusion rates of uranium into nickel and nickel into uranium.

The rate of growth of the total layer of intermetallic compounds was determined, as a function of time and temperature, over the range 300°-600°C. The results showed that at 500° and 600°C. penetration of nickel into uranium is about four times more rapid than that of uranium into nickel.

The activation energy for the temperature range 400°-600°C. was determined as $a=17{\cdot}1$ k. cal.

Six different intermetallic phases were distinguished under varying conditions of time and temperature.

Solubilities of Metals in Liquid Zinc: Literature Review

M. W. NATHANS: 'A Survey of Metal Solubilities in Liquid Zinc.'

U.S. Atomic Energy Commission, Argonne Nat. Laby. Report ANL-5753, July 1957; 72 pp.

This comprehensive literature survey of binary zinc-rich phase diagrams was initiated to provide a background against which an assessment could be made of the possibility of developing a fuel-decontamination process by means of fractional precipitation from metallic-zinc solution.

The survey is in four sections and has four appendices. *Section A* lists the systems for which no liquidus data were found; *Section B* details systems in which a liquid-miscibility gap occurs; *Section C* covers simple eutectic systems; *Section D* reviews systems other than simple eutectics, for which liquidus data were available. The appendices are devoted, respectively, to a *bibliography* of 76 items, solubility curves, tabular data on solubilities and a summary of crystallographic data.

The information assembled on the zinc-nickel system is given below.

'The Zinc-Nickel System

'There are six solid phases at room temperature (*Metals Handbook, Amer. Soc. Metals*, 1948): α-solid solution of zinc in nickel, between 0 and about 20 at. % zinc, β′ (NiZn) between 47 and 52 at. % zinc, γ′ (including $NiZn_3$) between 72 and 76 at. % zinc, γ (including the compositions $NiZn_4$ and Ni_5Zn_{21}) between 77 and 85 at. % zinc, δ at about 88 at. % zinc, and a terminal zinc phase ($NiZn_8$?). At elevated temperatures β′ decomposes peritectoidally into α+β, whereas β decomposes eutectoidally into β′+γ. γ′ decomposes peritectoidally into β′+γ. The distinction between γ′ and γ may be artificial.

'There are two peritectics:

L+α⇌β at 1040°C. and 52·3 at. % zinc.

L+γ⇌δ at 490°C. and 99·1 at. % zinc.

'The liquidus curve has a maximum at 882°C. and about 75 at. % zinc.

'Between 75 and 99·1 at. % zinc the γ phase is the product of primary crystallization, and between 99·1 and 100 at. % zinc it is the δ phase. At room temperatures the phases γ, δ and zinc may occur after non-equilibrium solidification of a melt containing more than 76 at. % zinc.

'The solubility curve is plotted in the report, from data tabulated by Heike *et al.* (*Metallwirtschaft*, 1933, vol. 12, p. 115). The data are listed in the table below. The γ phase (Ni_5Zn_{21}) is cubic, possibly body-centred, with $a_\circ=8{\cdot}904$ Å. (*Metals Handbook, loc.cit.*; *Zeitsch. physik. Chem.*, 1931, vol. 12 (B), p. 57.) The δ phase is hexagonal, a=8·873 Å, c=14·63 Å, c/a=1·649.'

Solubility of Nickel in Zinc

Temp. °C.	Nickel		Temp. °C.	Nickel	
	at. %	wt. %		at. %	wt. %
880·9	25·0	23	791	11·0	10
880·5	23·9	22	760	8·8	8
879	22·8	21	732	7·7	7
862	19·7	18	700-692	5·5	5
854	17·5	16	664	4·4	4
837	15·3	14	630	3·3	3
835	14·8	13·5	585	2·2	2
818	13·2	12	518	1·1	1
—	—	—	485	0·83	0·75

Aluminium-Manganese-Copper-Nickel Alloys

G. V. RAYNOR and C. R. FAULKNER: 'A Note on Aluminium-rich Aluminium-Manganese-Copper-Nickel Alloys.'

Jnl. Inst. Metals, 1957-58, vol. 86, Mar., pp. 323-4.

Following the discovery of the ternary compound $Ni_4Mn_{11}Al_{60}$, it was suggested that this compound might be regarded as analogous to $Cu_2Mn_3Al_{20}$ in the system aluminium-manganese-copper. The reasons for this suggestion are detailed in the paper.

X-ray work has confirmed that the crystal structures of $Ni_4Mn_{11}Al_{60}$ and $Cu_2Mn_3Al_{20}$ are indeed related, and in view of the similarities observed, it was considered advisable to define the relationship between the two ternary phases in the system aluminium-manganese-copper-nickel. The results of a limited survey of the quaternary are reported in this note.

Metallographic examination of slowly-cooled alloys and of alloys annealed for long periods at 500°C. indicates that the two ternary compounds in question form a complete series of solid solutions in the quaternary system.

Nickel-Titanium-Carbon Alloys

The following abstract is published of a paper presented at The Institute of Metals Division of The Metallurgical Society of The American Institute of Mining Engineers.

E. R. STOVER and J. WULFF: 'Nickel-Titanium-Carbon System.'
Jnl. of Metals, 1958, vol. 10, Feb., p. 100.

'An 870°C. isothermal section, the ternary solidus equilibria, and the solubility of titanium carbide and graphite in the nickel solid solution have been determined with quenched, arc-cast specimens. Each of the nickel-titanium binary phases forms two-phase fields with titanium carbide. Solution of carbon in Ti_2Ni increases the solidus temperature, the lattice parameter, and the microhardness. The effects of carbon solubility in $TiNi_3$ were much smaller, and no effects could be detected in TiNi. No solution of nickel in titanium carbide was found.

'A quasi-binary eutectic occurs between titanium carbide and the nickel solid solution at 13 at. per cent. titanium, 4 at. per cent. carbon, and 1307°C. On either side of the quasi-binary, ternary eutectics occur, with graphite at 1270°C., and with $TiNi_3$ at 1295°C. The nickel-graphite eutectic temperature was found to be 1328°C., and the nickel-$TiNi_3$ eutectic temperature was confirmed at 1304°C.

'The atomic per cent. solubility in nickel of titanium carbide together with graphite was determined from the Curie points of quenched samples, between 1260°C. and 600°C. The nickel corner of the three-phase field occurs at 3·4 per cent. titanium and 2·6 per cent. carbon at 1260°C., 3·1 per cent. titanium and 1·2 per cent. carbon at 1000°C., and less than 2·0 per cent. titanium and 0·2 per cent. carbon at 600°C. Thus, the binder of a titanium-carbide/nickel cermet must contain an excess of titanium, or some other carbide-forming element, if graphite precipitation is to be avoided during the sintering process.'

If and when this paper is published in full, notification will be made in *The Nickel Bulletin*.

Lattice Spacings and Structures of Metals

See abstract on p. 123.

Resistance of Nickel-containing Materials to Corrosion by Amine-Gas-Treating Solutions

See abstract on p. 146.

Fatigue Properties of Beryllium-Copper

See abstract on p. 148.

Effect of Irradiation on Resistance of Aluminium-Nickel Alloy to Corrosion by High-Temperature Water

See abstract on p. 148.

'Monel' Equipment for Handling Fluorine

See abstract on p. 149.

Determination of Chromium in Nickel

See abstract on p. 127.

Recovery of Metals from Alkaline Cyanide Solutions by Ion Exchange

See abstract on p. 125.

Classification of Filler Metals Conforming to A.W.S./A.S.T.M. Specifications

See abstract on p. 124.

NICKEL-IRON ALLOYS

Solubility of Nitrogen in Iron-Nickel Alloys

V. C. KASHYAP and N. PARLEE: 'Solubility of Nitrogen in Liquid Iron and Iron Alloys.'
Trans. Met. Soc., Amer. Inst. Mining Engineers, 1958, vol. 212, Feb., pp. 86–91; *T.P.* 4626*C*.

After brief review of the literature, the authors report experiments made to determine the solubility of nitrogen in liquid iron, binary alloys of iron with nickel, molybdenum and vanadium, and a ternary iron-molybdenum-vanadium alloy. The binary systems investigated contained, respectively, nickel 1–20, molybdenum 1–10, vanadium 1–10, per cent. The nine alloys in the ternary group contained 1, 2 or 3 per cent. vanadium and 1, 3 or 5 per cent. molybdenum. Solubility measurements were made at temperatures of 1600°–1800°C. Results are summarized in tables and graphs.

The results for iron confirm that Sievert's law is obeyed over the pressure range 50–750 mm. Measured solubilities and temperature coefficients determined by the authors are found to be in general agreement with those reported by other investigators in cases where comparable data are available. The equations used to assess the activity coefficient of nitrogen in the binary alloys are presented and details are given of a method by which the solubility of nitrogen in the iron-molybdenum-vanadium alloys was theoretically calculated.

It was found that in the iron-nickel alloys solubility of nitrogen decreased with rising nickel content. (Deviation from this behaviour in alloys containing up to 2 per cent. of nickel may possibly indicate an opposite trend at very low nickel contents, but since the accuracy of the relevant data is open to question, the authors consider that these findings should be ignored.)

Temperature has no effect on solubility in alloys containing 2 per cent. or more of nickel. The authors' results show good agreement with those obtained by WENTRUP and REIF in the temperature range 1600°–1700°C. (*Archiv f.d. Eisenhüttenwesen*, 1949, vol. 20, pp. 359–62; *Nickel Bulletin*, 1950, vol. 23, No. 2, p. 34).

Nickel-Aluminium-Cobalt-Iron Magnets: Development and Properties

W. ZUMBUSCH: 'The Development of 'Alnico'-Type Magnets containing Copper and/or Titanium. Properties of Currently Available Materials.'

Zeitsch. f. Metallkunde, 1958, vol. 49, Jan., pp. 1–8.

A review of the stages in development by which the nickel-aluminium-iron-base magnet alloys discovered by Mishima have been improved, to give the properties available in modern alloys of the 'Alnico' and related types. Particular attention is directed to modification of magnetic quality resulting from additions of copper and/or titanium, and from the use of specialized thermal treatments, including precipitation hardening and the application of a magnetic field in controlled-cooling conditions. Compositions and properties typical of alloys produced at various stages of development are listed. Reference is made also to magnet materials produced by sintering of powders of the component metals, and to the use, in special cases, of magnets of barium ferrite.

Magnetic Tape Produced by Powder Metallurgy

C. E. RICHARDS: 'British Achievements with Magnetic Tape Rolled from Powdered Metals.'

Electrical Manufacturing, 1957, vol. 9, Dec., pp. 104–8.

The author, who is associated with the Post Office Engineering Research Station, reviews some recent British work on development of powder-metallurgy production of magnetic alloys for use in electronic components. The paper deals specifically with work carried out with the dual aim of producing materials for very-thin-gauge, magnetically soft strip and tape, not only capable of giving acceptably high performance but also characterized by consistent reproducibility. This second factor is of major practical importance, since the performance of the magnetic materials is considerably affected by variation in composition, by the presence of trace impurities, and by other variables inevitably involved in conventional melting, casting and rolling procedures. (In this connexion, attention may be directed to earlier papers on co-operative work done by the Post Office Engineering Research Station and The Mond Nickel Company, Limited, on the production of ferromagnetic materials by powder-metallurgy methods: see *Amer. Inst. Electrical Engineers' Conference on Magnetism and Magnetic Materials*, 1956, pp. 131–3 and 134–7; abstracts in *Nickel Bulletin*, 1957, vol. 30, No. 6, pp. 93–4.)

After emphasizing the difficulties involved in achieving optimum magnetic properties in alloys produced by conventional melting, casting and fabrication methods, the author urges the advantages of applying powder-metallurgy techniques in this field. Brief notes are given on the basic processes used in production of strip from magnetic alloys and, in this connexion, data are tabulated to illustrate the ease with which composition can be controlled in the powder-made materials. In discussing the heat-treatment of the magnetic materials, mention is made of the relative immunity of the powder-made alloys to the presence of moisture during annealing. Methods of treatment used to ensure optimum properties are also briefly considered. Attention is directed to factors which must be taken into consideration in order to ensure optimum values of permeability and its temperature coefficient, and reference is made to the properties of some recently-developed magnetic alloys produced by powder-metallurgy techniques, e.g., 65–35 domain-oriented, 50–50 non-oriented, and 50–50 grain-oriented types of nickel-iron alloy.

A résumé is made of recent advances in magnetic materials for switching and storage devices, with notes on the switching characteristics of some powder-made alloys of improved types. Suggestions are made for methods by which still further improvement may be obtained in such materials.

Finally, the author summarizes the test procedure stipulated in Defence Specification D.E.F. 5192 for evaluation of the properties obtainable in the most recently-developed alloys, and notes some improvements in design of electromagnetic equipment to which such materials have contributed.

See also

'Cold Rolling Mill for Very Thin Strip.'
Engineering, 1958, vol. 205, Feb. 21, pp. 272–4.

The work described has a direct bearing on that reported in the preceding paper, since it gives details of the methods devised at the Post Office Engineering Research Station to modify a standard two-high rolling mill, used in conjunction with a simple insert mill, to permit production rolling of nickel-iron magnetic tapes to thicknesses of about 0·0001 in. (0·0025 mm.). Alternative types of mill are discussed, and consideration is given to the factors which caused them to be rejected as a basis for development. Finally, the main features of the modified mill are described (with details of spooling and tensioning equipment), its capabilities and limitations are discussed, and attention is drawn to patent rights and availability. The article includes data on a range of nickel-containing materials which have been successfully rolled on the mill.

Nickel and Nickel Alloys: Handbook

See abstract on p. 124.

CONSTRUCTIONAL STEELS

Inter-Relationship of Fatigue Resistance and Tensile Strength in High-Tensile Steel

J. H. CHILDS and M. M. LEMCOE: 'Fatigue Investigation on High-Strength Steel.'

Wright Air Development Center Report 56-205, July, 1957; 37 pp. *P.B.* 131371.

The report gives results of a comparison of variation in fatigue properties (especially endurance limit)

as a function of tensile strength in a nickel-chromium-molybdenum steel, heat-treated to three strength levels:

p.s.i.	*t.s.i.*	*kg./mm.²*
190,000	85	134
260,000	116	183
300,000	133	211

The composition of the steel used was carbon 0·395, silicon 0·31, manganese 0·75, sulphur 0·016, phosphorus 0·008, nickel 1·78, chromium 0·84, molybdenum 0·23, per cent. Tests were made by the increasing-load method (similar to Prot's method) in axial loading, to determine the variation in fatigue properties occurring under each of three mean stress conditions: zero, 60,000 and 90,000 p.s.i. (27 and 40 t.s.i.; 42·5 and 63 kg./mm.²). Conventional S-N curves were determined, by axial-load tests at 190,000 p.s.i. (85 t.s.i.; 134 kg./mm.²) ultimate tensile strength, under zero mean stress and 90,000 p.s.i. mean stress, to provide factors for adjusting the Prot-type failure stresses to standard mean endurance-limit values. In addition, Prot rotating-beam tests were made at 190,000 p.s.i. ultimate tensile strength.

The endurance limit under completely reversed axial stress increased with rise in ultimate tensile strength from 190,000 p.s.i. to 260,000 p.s.i., but appeared to decrease when the same material was heat-treated to the 300,000 p.s.i. level. Under high mean tensile stress the fatigue strength of the 300,000 p.s.i. material may be superior to that of the same material at the 260,000 p.s.i. level.

High-Tensile Nickel-Alloy Steel in Chamber for Testing Nuclear-Powered Aircraft Engine

'How the 'CANEL' Project Test Chamber was made.'
Iron Age, 1958, vol. 181, Feb. 27, pp. 106–7.

The article briefly discusses the advantages derived from the use of Lukens 'T-1' steel for construction of the chamber to be used in testing the nuclear-powered aircraft engine being developed under the Connecticut Aircraft Nuclear Engine Laboratory (CANEL) project. The test chamber, about 40 ft. (12 m.) long and 12·5 ft. (4 m.) in diameter, had to be designed to operate at high temperatures and pressures, and it is estimated that if carbon steel had been used, the total weight would have been more than 250 tons (254,000 kg.). By use of the high-tensile alloy steel, weight has been reduced to about 160 tons (162,550 kg.), and total cost has also been lowered.

The article includes a comparison between the mechanical properties of the 'T-1' steel and those of carbon steel of firebox grade. Attention is also directed to the good welding qualities of this type of alloy steel, and some details are given of heat-treatment and of welding procedure employed. Chemical composition of 'T-1' steel is quoted as carbon 0·10–0·20, silicon 0·15–0·35, manganese 0·60–1·00, sulphur 0·040 max., phosphorus 0·035 max., nickel 0·70–1·00, chromium 0·40–0·80, molybdenum 0·40–0·60, vanadium 0·03–0·10, copper 0·15–0·50, boron 0·002–0·006, per cent.

Resistance of Nickel-containing Steels to Corrosion by Amine-Gas-Treating Solutions

See abstract on p. 146.

Chemical Smoothing of Steel

'The A.R.D.E. Chemical-Smoothing Process for Steel. Some Recent Applications.'
Electroplating and Metal Finishing, 1958, vol. 11, Mar., pp. 82–4.

The chemical-smoothing process for steel developed at the Armament Research and Development Establishment, Woolwich, was first described by MARSHALL (*Jnl. Electrodepositors Tech. Soc.*, 1952, vol. 28, pp. 27–46; *Nickel Bulletin*, 1952, vol. 25, No. 4, p. 96).

The composition of the solution (which removes mild steel at the rate of approximately 0·4 mil/hour) is as follows (g./L.): oxalic acid (cryst.) 25, hydrogen peroxide 13, sulphuric acid 0·1. It is used at room temperature. The life of the solution is limited: one gallon (4·5 L.) is sufficient to treat 2·5 sq. ft. (0·2 sq. m.) of steel, based on a treatment time of one hour, and the mixture should be freshly prepared for each batch of work. In addition to its efficacy, the treatment has the dual advantage of low cost and ease of operation, and in recent years it has found expanding application. Some typical uses are briefly reviewed in this article, on the basis of information contained in a brochure issued by the Armament Research and Development Establishment. The process is not patented and advice on its use may be obtained from A.R.D.E.

Brief notes are given on the procedure used and facilities required for the smoothing treatment, and reference is made to its use in the salvage of rusted steel stores; reduction of friction in moving machine parts; smoothing of the inner surfaces of enclosed vessels; reduction, to specification, of slightly over-sized components; smoothing of mild steel prior to resistance welding; simultaneous smoothing and etching of metallographic specimens, and preparation for electroplating. In connexion with the last application, it is claimed that the preliminary smoothing promotes very strong adhesion of thick nickel electrodeposits, and, since the smoothed steel is covered with a thin passive film which prevents further oxidation, a delay of as much as 8 minutes before insertion in the plating bath has been found to have no adverse effect.

In electrodeposition of chromium prior smoothing of the basis steel reduces or eliminates formation of nodular growths.

Fracture Characteristics of Temper-Brittle Steel

L. D. JAFFE and D. C. BUFFUM: 'Fracture of Temper-Brittle Steel.'
Trans. Met. Soc., Amer. Inst. Mining Engineers, 1958, vol. 212, Feb., pp. 73–4; *T.N.404E.*

It has been known for some years that the path of brittle fracture in temper-brittle steel differs from that found in unembrittled steel, and more recently it has been recognized that alloy steels

may display the defect in two forms, which have been designated respectively 'upper-nose' and 'lower-nose' temper-brittleness. No experimental evidence has hitherto been published, however, to demonstrate that a differentiation can be made between the two forms of brittleness by the inspection of brittle fractures.

This paper compares the characteristics of fractures in two temper-brittle low-alloy nickel-chromium steels which had been treated, respectively, by the following cycles:—

(1) Austenitized for one hour at 900°C., tempered for one hour at 675°C. and embrittled for 48 hours at 500°C., with water-quenching after each treatment.

(2) Austenitized for one hour at 900°C., water-quenched, heated for 240 hours at 650°C., water-quenched.

Details are given also of the fracture of an unembrittled material from the same heat. In this case the heat-treatment had consisted of one hour at 900°C., water-quenching, and one hour at 675°C., followed by water-quenching. The treated steels were fractured, in all three cases, at temperatures and in conditions which would give rise to brittle fracture. The structures illustrated in the paper show that the path of fracture of the material embrittled in the lower-nose region (1) was mostly along austenite grain boundaries, whereas in material embrittled in the upper-nose region (2) the path of fracture was mostly along ferrite grain boundaries. In the nominally unembrittled material fracture is through the grains, by cleavage.

Classification of Filler Metals Conforming to A.W.S./A.S.T M. Specifications

See abstract on p. 124.

Nickel and Nickel Alloys: Handbook

See abstract on p. 124.

HEAT- AND CORROSION-RESISTING MATERIALS

Influence of Boron and Zirconium in High-Temperature Materials

R. F. DECKER and J. W. FREEMAN: 'Mechanism of Beneficial Effects of Boron and Zirconium on Creep-Rupture Properties of a Complex Heat-Resistant Alloy.'

Jnl. of Metals, 1958, vol. 10, Feb., p. 102.

The following abstract is published of a paper presented at the Institute of Metals Division of the Metallurgical Society of the American Institute of Mining Engineers.

'Recent research revealed that a major effect of melting practice on the creep-rupture properties of an alloy containing nickel 55, chromium 20, cobalt 15, molybdenum 4, titanium 3, aluminium 3, per cent., was through introduction of trace boron and zirconium into the alloy.

'This research has been continued by a microstructural investigation to establish the mechanism of the pronounced benefits of boron and zirconium on creep-rupture properties of the alloy at 1600°F. (870°C.).

'Materials with varying boron and zirconium content were exposed to creep conditions and the microstructures were analysed by optical and electron microscopy, electron diffraction, microfractography and hardness measurements. Particularly useful were interrupted creep tests, which allowed comparisons of materials after equivalent exposures.

'The mechanism of improvement of creep-rupture properties was found to be a pronounced stabilizing effect of boron and zirconium on the grain boundaries of the alloy. The alloy with low boron and zirconium was subject to rapid agglomeration of $M_{23}C_6$ and γ' in the grain boundaries, followed by depletion of γ' and intergranular micro-cracking at the grain boundaries transverse to applied stress. Brittle fracture then occurred by linking of micro-cracks. However, additions of zirconium, boron, and boron+zirconium decreased this tendency, in that order. In the absence of these elements extensive micro-cracking was found early in second-stage creep, at relatively short periods, and fracture occurred prematurely, with very little deformation. Proper amounts of boron+zirconium delayed micro-cracking until after third-stage creep had started, so that creep-rupture life was greatly prolonged and ductility-to-fracture markedly increased.

'No effect of the trace elements on the size, amount, and distribution of the intragranular γ' was detected. Accordingly, the property effects were not found to result from a change in the intragranular γ' reaction.'

If and when this paper is published in full, notification will be made in *The Nickel Bulletin.*

Nickel-Titanium-Carbon Alloys

See abstract on p. 134.

Effects of Forging Temperature and Heat-Treatment on Performance of Gas-Turbine Blading

C. A. GYORGAK, J. R. JOHNSTON and J. W. WEETON: 'Performance of 'Inconel 550' Turbine Blades in a Turbojet Engine and Effects of Different Forging Temperatures and Heat-Treatments.'

Nat. Advisory Committee for Aeronautics, Research Memorandum RM E55F08, Aug. 16, 1955; 55 pp.

The investigation described was planned to evaluate 'Inconel 550' as a potential material for turbine blading. (In this alloy, which is essentially a modification of the nickel-chromium-base precipitation-hardenable 'Inconel X', the aluminium content is increased from 0·7 to 1·2 per cent. The stress-rupture properties of the '550' grade lie in the upper region of the range published as typical for 'Inconel X'.

The specific aim of the work was to determine the effects, on performance of 'Inconel 550' blades, of variations in (1) forging temperature, and (2) times and temperatures of solution and ageing treatments.

Seven blades from each group were randomly selected for engine tests, and eight blades made from 'S-816' (an alloy which has been used in the U.S.A. for turbine blades) were employed as controls. The test blades of both materials were run to failure under cyclic conditions (15 minutes at rated speed of 11,500 rev./min. and 5 minutes at idle speed of 4,000 rev./min.): temperature of operation 1500°F. (815°C.). The stress-rupture lives of specimens cut from blade aerofoils were determined, at the same temperature, under stresses of 20,000, 25,000 or 30,000 p.s.i. (9, 11 or 13·5 t.s.i.; 14, 17·5 or 21 kg./mm.^{2}), and were compared with data from bar stock taken from the same heat of material. Metallurgical studies were made of blades in the as-heat-treated conditions, and after engine operation.

The results are discussed at some length, and consideration of the metallographic observations is supplemented by reference to numerous photomicrographs. The conclusions drawn from the various series of tests are summarized below:

'Best engine performance was associated with a forging and solution temperature of 2150°F. (1175°C.). Reduction of the temperature for forging or solution treatment to 1950°F. (1065°C.) gave shorter blade life, but it appeared less damaging to lower the forging temperature to 1065°C. if the solution treatment was done at a temperature above 2100°F. (1150°C.) than to lower the solution temperature to 1950°F. (1065°C.) after forging at 2150°F. (1175°C.).

'Life of the best group of 'Inconel 550' blades ranged from 450 to 857 hours: the 'S-816' stock blades had lives of 204 to 599 hours.

'Fatigue characteristics were associated with almost all 'Inconel 550' and 'S-816' blade failures.

'Blade performance could not be correlated with grain size, but best performance was obtained from the group of blades having the largest grain size and the greatest scatter in this variable, even though fatigue was associated with the failure. Both fine and coarse grains were found in most blades, but no 'elephant' grains were found.

'Engine performance could not be correlated with the microstructure of the alloy: differences from blade to blade and within a single blade were greater than those found among the various groups of blades. Microstructures after engine operation appeared to be generally the same as before operation: the only differences noted were more rapid response to etchants after engine testing, and the appearance of depletion zones in the outer surfaces of these blades.

'The highest hardness of heat-treated blades was associated with solution-treatment temperatures of 1950° and 2100°F. (1065° and 1150°C.). Increasing the degree of solution (by treatment at 2150°F. (1175°C.) had a tendency to lower the hardness obtained after ageing, but permitted increase in hardness to occur during engine operation.'

Effect of Hot Working on Structure and Properties of High-Temperature Materials

The following abstract is published of a paper presented at the Institute of Metals Division of the Metallurgical Society of the American Institute of Mining Engineers.

A. P. COLDREN, J. W. FREEMAN and C. B. HARTLEY: 'Effect of Hot Working on the Structure and Creep-Rupture Properties of Heat-Resistant Alloys.'
Jnl. of Metals, 1958, vol. 10, Feb., p. 97.

'Hot working can be used to vary the creep-rupture properties of alloys in the as-worked condition over wide ranges. Data have been obtained on commercially pure nickel ('A' Nickel), indicating that during isothermal hot working the per cent. reduction has a much greater influence on properties at 1100°F. (595°C.) than does the temperature of working, provided that recrystallization does not occur during working or testing. The results do not correlate perfectly with simple measures of strain hardening. Preliminary structural studies indicate a likely, added influence of substructures.

'Certain alloys show a pronounced effect of hot-working conditions even when solution-treated and aged after hot working. Data presented for 'A-286' alloy, in the solution-treated and aged condition, show the influence of prior hot-working temperature and reduction under isothermal conditions. For example, a ten-fold variation in the rupture life at 1200°F. (650°C.) and 65,000 p.s.i. (29 t.s.i.; 45·5 kg./mm.^{2}) was effected by variations in prior working conditions. Preliminary results of structural studies to identify the mechanism are given.

'The hot-working conditions employed in this investigation cover the usual ranges of temperature and reduction, which usually involve working below the temperature of simultaneous recrystallization.

'The present study is therefore not confined to temperatures at which recrystallization occurs during working.'

If and when this paper is published in full, notification will be made in *The Nickel Bulletin*.

Effects of Overheating on Aircraft Gas-Turbine Blading Alloys

Investigation of the effects of intermittent overheating has been studied on two alloys typical of materials developed for the blading of aircraft gas turbines: see following abstracts.

(1) J. P. ROWE and J. W. FREEMAN: 'Effect of Overheating on Creep-Rupture Properties of 'S-816' Alloy at 1500°F. (815°C.).'
Nat. Advisory Committee for Aeronautics, Tech. Note 4081, Dec. 1957; 71 pp.

The material used was a wrought alloy reported to be of the following composition: carbon 0·42, chromium 20·00, nickel 20·40, molybdenum 3·93, tungsten 3·57, niobium 3·50, iron 3·90, manganese 1·37, silicon 0·58, per cent., balance cobalt.

The tests were designed to evaluate the effects of

brief repeated overheating, to temperatures of 1650°, 1800°, 1900° and 2000°F. (898°, 982°, 1038° and 1093°C.), on the creep-rupture properties of the alloy at 1500°F. (815°C.), under stresses within the range of its rupture strengths for 100–1000 hours.

Possible damage from overheating was considered to include internal changes in the structure of the metal induced by exposure to the higher temperatures, and loss in life by creep if stress was present during overheat. Temperature damage was evaluated by starting the tests at 1500°F. (815°C.) and periodically overheating with the stress removed during the overheating periods: stress damage during overheat was assessed by leaving the specimens under stress during overheat. Overheat periods were in most cases of 2 minutes' duration, occurring cyclically at approximately 5- or 12-hour intervals. (The conditions were chosen to simulate those liable to occur in jet-engine operation.)

The experimental results are reported at length. They led to the following main conclusions:

'1. Overheating at temperatures up to 2000°F. (1093°C.) reduced the rupture life of the alloy at 1500°F. (815°C.). The degree of loss in life increased with both rise in temperature and accumulated time of overheating. The only exception was some increase in rupture life from limited overheating to 1600° and 1650°F. (870° and 900°C.) in the absence of stress.

'2. Loss in rupture strength arises from both alteration of structure by temperature effects and, if stress is present, by temperature acceleration of creep. The presence of appreciable stress during overheating would be the predominant cause of damage. Even very brief exposure at 1800°–2000°F. (982°–1093°C.) under stresses normally causing rupture in 100 - 1000 hours at 1500°F. (815°C.) would either exhaust a large proportion of the rupture life or cause immediate rupture.

'3. Temperature alone can reduce rupture times at 1500°F. (815°C.) to a pronounced extent. Overheating at 2000°F. (1093°C.) for two minutes caused a severe drop in life: at lower temperatures the effects were less drastic, even for a large number of overheats.

'4. It is possible to compute reasonably well the combined effects of temperature and creep damage caused by overheating in the presence of stress. Loss in life from temperature cycling must be added to losses by creep. Estimation of damage resulting from overheating requires prior measurement of the effect of overheat temperature on time of rupture at 1500°F. (815°C.). The creep damage can be estimated as the per cent. of total available rupture time at the overheat temperature represented by the actual time at that temperature.

'5. Temperature alone induces internal structural changes which influence strength at 1500°F. (815°C.), but results obtained in creep tests at that temperature on material which had been subjected to only two overheats, each of 2 minutes' duration, in the range 1650° - 2000°F. (898° - 1093°C.), showed that the effect of one or two overheats becomes significant only when the temperature is 1900°F. (1038°C.) or above.

'6. Cyclic overheating, with stress removed during the overheats, until fracture occurred at 1500°F. (815°C.), disclosed that a saturation in damage occurred for temperatures of 1650°, 1800° and 2000°F. (898°, 982° and 1093°C.). After a specific number of overheats no further damage occurred. With overheating at 1900°F. (1038°C.) saturation was not obtained.

'7. Microstructural studies indicated that overheating to 1650°F. (898°C.) and 1800°F. (982°C.) accelerated the normal precipitation and agglomeration process which occurs during testing at 1500°F. (815°C.). It is believed that heating to 1900°F. and 2000°F. (1038° and 1093°C.) caused part of the alloying elements which participate in normal precipitate formation during test at 1500°F. (probably carbon and/or nitrogen) to be transferred to inactive niobium carbonitrides. This reaction reduces formation of precipitate during tests at 1500°F. (815°C.) and, if the total time of overheating at 2000°F. (1093°C.) is sufficient for the reaction to be completed, there is the appearance of a solution-treated structure. This, however, does not apparently account for the very pronounced loss in strength resulting from brief overheating at 2000°F. (1093°C.). The main loss in rupture life occurred before the period of overheating and was sufficiently long to cause a recognizable change in the microscopically visible structure of the alloy. This observation suggests that there is probably a sub-microscopic coherent precipitate containing carbon and/or nitrogen which provides high strength, and that this precipitate is destroyed even by very short overheating at 2000°F. (1093°C.).

'8. Repeated cyclic overheating causes more extensive and more rapid temperature damage than results from heating the material to the same temperatures before testing. It is therefore not possible to predict temperature damage by overheating before testing.

'9. A limited number of overheats at any time during the creep-rupture life apparently has about the same effect as if they were applied early in the test.'

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(2) J. P. ROWE and J. W. FREEMAN: 'Effect of Overheating on Creep-Rupture Properties of 'HS-31' Alloy at 1500°F. (815°C.).'

Nat. Advisory Committee for Aeronautics, Tech. Note 4083, Dec. 1957; 78 pp.

The material used in this investigation (known as 'HS-31' or 'X-40' alloy) was a precision-cast alloy of the following composition: carbon 0·50, chromium 25·89, nickel 10·52, tungsten 7·50, iron 1·25, manganese 0·55, silicon 0·72, phosphorus 0·016, sulphur 0·008, per cent., balance cobalt.

Temperatures and test conditions were closely parallel with those used in the tests on 'S-816' alloy:

see preceding abstract. In the tests on 'HS-31' alloy the stresses used at 815°C. were within the range of rupture strengths of the material for about 100-700 hours.

The conclusions to be drawn from the tests are in most respects similar to those reported for the 'S-816' alloy. Similarities and differences are indicated below.

1. General conclusion as for 'S-816'. The only exception to the deleterious effect of overheating was, in this case, some increase in rupture life from limited overheating at 1600°, 1650° and 1800°F. (870°, 900° and 980°C.) in absence of stress.

2. Conclusions as for 'S-816' (*see* conclusion 2 of preceding abstract).

3. In this case also temperature alone reduced rupture times at 1500°F. (815°C.). Two overheats, each of 2-minutes' duration, at 2000°F. (1093°C.) halved rupture time at 1500°F. (815°C.), but the effects were less severe for overheats at lower temperatures.

4. Conclusions as for 'S-816' (*see* conclusion 4 of preceding abstract).

5. Conclusions as for 'S-816' (*see* conclusion 5 of preceding abstract).

6. In this alloy also overheating at 1650°-2000°F. (898°-1093°C.) accelerated agglomeration of precipitates which normally form during testing at 1500°F. (815°C.).

7 and 8. *See* conclusions 8 and 9 of preceding abstract.

Properties of Nickel-Chromium-base and Other High-Temperature Alloys

K. B. YOUNG: 'High-Strength Nickel-Chromium Alloys for Elevated Temperature Service.'

Canad. Aeronautical Jnl., 1957, vol. 3, Oct., pp. 287-95.

Although the elevated-temperature properties of nickel-chromium-base alloys, particularly their oxidation-resistance, have been successfully utilized for the past 50 years, e.g., in electrical-resistance elements, the prominence of modified types of these alloys for applications involving high stresses and high temperatures is of relatively recent origin, coinciding with the development of aircraft gas turbines. Components of this type of power plant still constitute the major field of application for such alloys, but they are being introduced also in other applications in which their outstanding qualities are essential. Prospects for continued use in gas-turbine units of still greater power must be considered in relation to possible developments in design, and to other factors.

Against this background the properties of various nickel-chromium-base alloys in bar, sheet, and cast form, are discussed by the present author. For the purposes of this paper, 100-hour rupture strength was selected as a criterion of comparison, but it is emphasized that the values given are not design minima and do not necessarily represent the order of merit of the respective materials when other properties also are taken into consideration.

The subject is discussed in the classifications shown below.

Historical Background

The author briefly traces the development of nickel-chromium-base alloys from the types in service before World War II to the advent of the gas-turbine engine and the intensive investigation of precipitation-hardenable alloys based on the 80-20 nickel-chromium system, with ultimate development of the 'Nimonic' series of alloys. Since the early 1940's, those alloys have been standard materials for aircraft gas-turbine blading in British production engines. (For more detailed information on alloys of the 'Nimonic' series, attention may be directed to papers abstracted in *Nickel Bulletin*, 1956, vol. 29, No. 12, p. 217; 1957, vol. 30, No. 7-8, p. 129.)

In the U.S.A., the nickel-chromium-base alloys 'Inconel W' and 'Inconel X' were developed to meet the requirements of that country, but their use as gas-turbine rotor blading was limited, and, during the war period, cobalt-base alloys, e.g., of the 'S-816' type, and 'Hastelloy' nickel-molybdenum-base alloys were widely used in American engines. In later years the high-temperature potential of the nickel-chromium alloy system was more fully realized in the U.S.A. and a range of alloys of that type has since been developed, e.g., 'Inconel 700', 'Waspaloy', 'M-252' and 'Udimet 500'.

Properties of Wrought Nickel-Chromium-(Cobalt) Alloys

The 100-hour rupture strengths of alloys of the 'Nimonic' and 'Inconel' series are presented to show, by correlation with the chemical compositions of the respective alloys, the beneficial influence of increased additions of the precipitation-hardening elements, aluminium and titanium, and of the matrix-strengthening element cobalt. Reference is made to problems of workability associated with development of the more refractory alloys of the series, and to advances made possible by introduction of the Séjournet glass-lubricated extrusion process.

Alloys in sheet form are discussed separately, since production and fabrication methods vary from those used for bar-stock material. Difficulties associated with fabrication and treatment of sheet material are considered and typical properties are illustrated by graphs showing scaling- and oxidation-resistance at various temperatures, and typical mechanical and physical properties. The data show that the nickel-chromium-base alloys (e.g., 'Inconel', 'Nimonic 75', 'Inconel X') are, in general, superior to the iron-base types. Attention is directed to the satisfactory short-time high-temperature tensile properties and the excellent oxidation-resistance of 'Inconel 702': it is suggested that the use of aluminium, as opposed to titanium, as the hardening medium, may be the factor responsible. Finally, a note is added on the significance, in relation to welding, forming, and machining operations of the alloys, of loss of ductility in the temperature range in which age-hardening occurs.

Properties of Nickel-Chromium-base Cast Alloys

Problems of forgeability have restricted the range of alloys which can be used in wrought form for high-temperature applications, and, with the continued requirement for materials which will withstand higher working temperatures, increased attention has been directed to the possible application of cast materials for parts such as rotor blades. Cast 'Udimet 500', 'GMR 235' and 'Inconel 713C' are representative of recent commercial developments in this field. The properties of these alloys are discussed and some reference is made to earlier investigations on 'Guy alloy' and 'Kinsalloy', cast materials which proved unsuitable for further development. Although the cast alloys are inferior to wrought types in ductility and impact strength, they probably have a useful future in high-temperature engineering. Some data are presented to illustrate the creep-rupture strength of 'Inconel 713C' and 'GMR 235', for which the values shown are superior to those of the cast cobalt-base alloy 'Stellite 31', and it is indicated that the thermal-fatigue resistance of 'Inconel 713C' is also better than that of 'Stellite 31'. In this connexion, the author refers to reports which have correlated response to thermal shock with oxidation-resistance: it is tentatively suggested that the high aluminium content in 'Inconel 713C' may, by its beneficial effect on resistance to oxidation, contribute to the exceptionally good thermal-shock resistance of the alloy.

Properties of Wrought Iron-base Nickel-Chromium-Iron Alloys

The mechanical properties of iron-base alloys, at temperatures higher than 1400°F. (760°C.), are too low to be of interest, but in the range 1000°-1400°F. (540°-760°C.) 'Incoloy'-type alloys are not only equivalent or superior to higher-nickel alloys, but also have the advantage of lower cost and easier fabrication. Creep-rupture curves compare the strengths of typical iron-base turbine-disc alloys, 'Discalloy', 'A-286' and 'Incoloy 901'.

Influence of Heat-Treatment

The significance of heat-treatment, in producing optimum properties in nickel-chromium precipitation-hardening alloys, is outlined by reference to the effect of solution and ageing treatments on fatigue and creep properties, grain size, and the form and quantity of precipitate occurring in the aged material. Creep and fatigue curves for 'Inconel 700' illustrate the effect of thermal treatments which vitally influence these variables, indicating the possibility of achieving desired combinations of properties by means of selected cycles of treatment.

Influence of Composition

This section comprises a brief résumé of the type and effect of the phases precipitated in 'Nimonic' and 'Inconel' precipitation-hardenable alloys. Reference is made also to the influence, deleterious or beneficial, of trace amounts of various elements.

Electroless Nickel as a Brazing Material

See abstract on p. 131.

Nickel and Nickel Alloys: Handbook

See abstract on p. 124.

Titanium-Carbide-base Cermets: Engineering Properties

J. C. REDMOND, R. J. REINTGEN, J. R. FENTON and M. E. SIMON: 'Evaluation of the Engineering Properties of Titanium-Carbide-base Cermets.'

Wright Air Development Center Tech. Report 57-25, July 1956; 60 pp. *P.B.* 131026.

This report is the most recent in a series giving details of work by Kennametal Inc., which has been in progress for several years, with the aim of obtaining comprehensive information on the properties of titanium-carbide-base cermets. The specific object of the study is the assessment of the suitability of such materials for use in gas-turbine components or other high-temperature applications. (Results of earlier work were contained in *W.A.D.C. Tech. Reports* 52-235 and 55-100.)

The binders used in the materials examined were nickel, or high-nickel alloys of the following types: nickel-molybdenum, nickel-(molybdenum)-aluminium-chromium, nickel-molybdenum-aluminium-($TiSi_2$), nickel-molybdenum-aluminium-iron-cobalt, and nickel-chromium-tungsten. One composition was of iron base and contained nickel, molybdenum and aluminium. The nickel content of the cermets varied from 17·5 to 60 wt. per cent. and the titanium carbide from 27·5 to 60 wt. per cent.

The report gives details of equipment and procedure used in a new type of short-time tensile test by which stress-strain data can be obtained at temperatures varying from normal to 1800°F. (980°C.), and above. The test was originally conceived as a 'seeding' method for rapid determination of the high-temperature strength and ductility of newly developed materials, with a view to deciding whether their properties were sufficiently high to warrant further investigation by stress-rupture and other tests. Comparison of test data so obtained, with those derived by other methods showed, however, that the test is also useful *per se*, and since there were indications that it might be of use in predicting impact characteristics, its results were correlated with those of impact tests.

Selected carbide/metal compositions were subjected to stress-rupture tests at 1600° and 1800°F. (870° and 980°C.); also to Charpy and ballistic-impact tests, and to fatigue tests at room temperature, at 1600° and at 1800°F. (870° and 980°C.). In addition, the effect, on the properties of the composites, of the method by which they had been made was investigated (i.e., extrusion *vis-à-vis* cold-pressing and sintering).

All the cermets tested showed a measurable degree of ductility: particular reference is made to materials in which a nickel-chromium-tungsten-alloy binder

was used: at 1600°F. (870°C.) and above these materials showed an elongation approaching 3 per cent. A series of cermets containing a nickel-molybdenum - aluminium - chromium - alloy phase, under stress-rupture test, showed 100-hour strengths of 50,000 p.s.i. (22·5 t.s.i.; 35·2 kg./mm.²) at 1600°F. (870°C.), and 18,000 p.s.i. (8 t.s.i.; 12·5 kg./mm.²) at 1800°F. (980°C.). These are the highest strengths so far obtained with this type of cermet. The nickel-chromium-tungsten/titanium-carbide cermets also exhibited a useful degree of high-temperature strength, in addition to their toughness. The stress-rupture properties of cermets made by extrusion were at least equal to those produced by the conventional cold-press-and-sinter method.

Fatigue tests confirmed that, as a class, these cermets have excellent properties, showing values superior to those typical of high-temperature alloys. Binder content appeared to have little effect on fatigue behaviour. No direct relation was found between fatigue strength and stress rupture.

Impact tests on some of the more ductile compositions (e.g., those containing nickel-chromium-tungsten as the alloy phase) showed properties approaching those characteristic of some of the high-temperature alloys at present in service. It was found also that these types of cermet, when subjected to ballistic-impact tests, did not fail catastrophically and that damage was restricted to a small area near the point of impact.

Cermets: Bibliography of Recent Literature

See abstract on p. 124.

Oxidation-Resistant Coatings for Molybdenum

Operation of gas turbines is at present limited to a maximum temperature of about 1600°F. (870°C.), primarily by the lack of materials of construction suitable for use at higher temperatures. Future developments may eventually entail temperatures of the order of 2500°F. (1370°C.) and service conditions involving also long-term exposure to severely oxidizing atmospheres. Molybdenum and certain of its alloys are at present the only readily available materials having the mechanical properties which would be adequate in such applications. At 1600°F. (870°C.) molybdenum alloys have better strength than any other known alloy and this superiority is even more marked at higher temperatures, but, due to the 'catastrophic' oxidation to which molybdenum is subject at these temperatures, it is at present impossible to utilize to full advantage the mechanical strength of the metal.

Intensive research on development of molybdenum-containing oxidation-resisting alloys has, until now, been of little avail, since any increase in oxidation-resistance has been at the expense of mechanical properties. (See for example, papers by SPRETNAK and by RENGSTORFF, abstracted, respectively, in *Nickel Bulletin*, 1956, vol. 29, No. 3, p. 45, and No. 5, p. 93.) Current efforts are therefore concentrated on development of suitable protective coatings. The reports abstracted below give details of two such investigations.

A. C. WERNER, J. J. SHYNE, H. N. BARR and B. TRIFFLEMAN: 'Preparation of Protective Coatings by Electrophoretic Methods.'

Wright Air Development Center Tech. Report 56-251, Apr. 1957; 46 pp. *P.B.* 131062.

As an introduction, the authors list five requirements which must be satisfied by coatings which could be useful for protection of molybdenum. It is concluded that the coating must be absolutely impervious; it must be resistant to severe thermal shock; it must withstand erosion by dust and other particles; the properties must be retained at high temperatures when the molybdenum is under stress; and, because failure of one blade or other component may be vital for the safety of the aircraft, it is also essential that the coatings be reproducible and that defective coatings be easily detected by a reliable test.

Since earlier experiments had shown that 80-20 nickel-chromium alloy most nearly satisfied these requirements, this composition was chosen as the main coating material investigated in the series of tests now reported. The particular features of the electrophoretic method which led to its use for deposition of the coatings were the possibility of plating multilayer uniform coatings of closely-controlled thickness, on articles of irregular shape, and the fact that it also offered a means of depositing cermet coatings, which would give maximum resistance to erosion. It is a further advantage that more closely-packed coatings are producible by this method than can be obtained by brushing or spraying. The procedure used to deposit the coatings is described in detail. The first stage is the deposition, under the influence of a potential gradient, of charged particles suspended in a liquid. These deposits are subsequently densified by isostatic pressing or ball-peening, and are finally sintered to form dense, adherent coatings. In the case of a metallic coating, adherence is usually improved by deposition of a reducible oxide and the introduction of a reduction stage before densification.

Four types of coating were investigated:—

(1) 80-20 nickel-chromium alloy, by deposition and reduction of NiO/nickel-chromium powder mixture.

(2) Chromium coatings, by deposition of fine-particle Cr_2O_3.

(3) A nickel-chromium/$MoSi_2$ cermet coating containing 50 vol. per cent. of $MoSi_2$ in an 80-20 nickel-chromium matrix.

(4) A nickel/Cr_3C_2 cermet coating containing 50 vol. per cent. of Cr_3C_2 in a nickel matrix.

Full details are given of methods of surface deposition of the coatings, and of the drying, reduction,

densification and sintering procedures used in the respective series.

To evaluate the coatings, static-air-oxidation, ballistic-impact, erosion and thermal-shock tests were made on panels to which 2–5 coating layers had been applied. (The maximum thickness which could be obtained in one coating was 3 mils.) In all cases the dense nickel-chromium layer was deposited first. (Attempts to use chromium coatings as diffusion barriers between the nickel-chromium-alloy layer and the molybdenum were unsuccessful.)

A three-layer coating consisting of Ni-Cr/Ni-Cr_3C_2/Ni-Cr withstood air oxidation for 500 hours at 1800°F. (980°C.) + a further 500 hours at 2000°F. (1095°C.). Metallographic examination showed extensive intergranular corrosion of the coating.

Fully densified coatings were found to be incapable of withstanding ballistic impact at 1800°F. (980°C.): coatings were therefore prepared in which the outer layer was undensified nickel-chromium, in one case protected by a cermet coating. The latter coatings proved satisfactory under impact.

Resistance to erosion was found to be a function of the rate of flow of the abrading medium. Some of the coatings withstood abrasion with Al_2O_3 at 1800°F. (980°C.) at 30 g./min., but failed at 60 g./min. It is considered that erosion-resisting quality can be assessed only on the basis of field results. In the thermal-shock tests, the coatings proved resistant to 200 rapid thermal cycles over the range 200°–1800°F. (95°–980°C.), but failed before completion of the specification of 400 cycles laid down as a criterion.

Recrystallization of molybdenum which occurred during deposition of the coatings is not considered important, since some molybdenum alloys have recrystallization temperatures above the sintering temperatures employed.

See also

D. V. DOANE: 'Oxidation-Resistant Coatings for Molybdenum.'

Wright Air Development Center Tech. Report 54-492, *Pt. III*, Apr. 1957; 94 pp. *P.B.* 131368.

This report is the last of a series of three. It contains, in addition to details of recent work, a summary of knowledge gained as the result of the comprehensive investigation covered by a six-year study.

Part I, issued in 1954, described equipment and procedures used in applying sprayed-metal coatings to molybdenum, and the tests used to evaluate the behaviour of the coated materials. The results of the tests emphasized the importance of processing control as a factor in obtaining oxidation-resistance by means of sprayed coatings. Low-stress coatings of electrodeposited chromium proved promising.

Part II, June 1955, gave details of ballistic-impact tests; summarized results of further study of variables involved in spray coating; gave results on composites incorporating sprayed coatings considered promising, and reported thermal-cycling tests on molybdenum coated, by electrodeposition, with chromium, nickel, and nickel + chromium. A study had also been made, at that stage, of changes in the nature of an aluminium-chromium-silicon-coating during exposure to air at 1800°F. (980°C.).

Research since the issue of Part II has varied widely in its objectives, and the latest Report, *Part III*, is therefore divided into nine sections, each of which deals with one specific aspect of the investigation. The scope of the work is indicated below, and the main conclusions drawn are summarized at the end of the abstract.

The basis metal used in the majority of the more recent tests was an arc-cast 0·5 per cent. titanium-molybdenum alloy, preferred to molybdenum because of its superior strength and its high temperature of recrystallization. In some sections of the investigation both unalloyed molybdenum and the molybdenum-base alloy were used. The term 'material' used in the abstract to designate the base coated indicates molybdenum and/or the alloy. For exact details the original must be considered.

Section 1. *Further Evaluation Tests on Coatings previously tested*

Various tests were made on coatings of chromium, and ductility and erosion tests were carried out on material coated with an aluminium-chromium-silicon alloy, a nickel-silicon-boron alloy, a nickel-chromium-boron alloy ('Colmonoy No. 5'), an aluminium-nickel-silicon alloy, and a composite coating consisting of 'Colmonoy No. 6' (nickel-chromium - boron alloy) + aluminium - chromium-silicon.

Section 2. *Modified Sprayed-Coating Compositions*

Data derived from the previous experiments had shown that no single coating exhibited outstanding resistance in both impact and thermal-shock tests at 1800°F. (980°C.), but that better performance was obtained with the composite nickel-chromium-boron + aluminium-chromium-silicon combination. In Section 2, therefore, further study was made of various composite coatings embodying, in varying combinations, nickel-base alloys, aluminium-bonded alloys, aluminium-chromium-silicon alloys of high aluminium content, and electrodeposited porous chromium coatings spray-coated with aluminium-chromium-silicon.

Section 3. *Electrodeposited Chromium/Nickel Coatings*

Oxidation, ductility, thermal-cycling, ballistic-impact and erosion tests were made on specimens electroplated first with chromium and subsequently with nickel.

Section 4. *Nickel-Clad Molybdenum Alloy*

Tests similar to those to which other specimens had been subjected were made not only on nickel-clad specimens, but on specimens which, after cladding with nickel, had been further protected by a sprayed coating of aluminium-chromium-silicon.

Section 5. Ductility of Coated Molybdenum Alloy at Room Temperature

Bend and impact tests were made to compare the ductility of the molybdenum alloy (1) as-rolled, (2) as-coated and diffusion-treated, using two sprayed coatings (aluminium-chromium-silicon and nickel-chromium-boron), and (3) after long exposure at 1800° and 2000°F. (980° and 1095°C.), with and without coating.

Section 6. Coatings for Service up to 3000°F. (1650°C.)

Under this heading the programme comprised three separate phases: (1) development of equipment for evaluation of oxidation-resistance at these higher temperatures, (2) screening tests on coatings which had given promising results in the range 1800°-2000°F. (980°-1095°C.), and (3) exploration of coatings considered promising for service at the higher temperatures envisaged. In this connexion data on the behaviour of nickel-chromium-boron and nickel-silicon-boron coatings are included.

Section 7. Barrier-Layer Coatings

Introduction of an intermediate coating of copper was considered a possible means of inhibiting diffusion between the molybdenum-base metal and the nickel-base coating. Such interdiffusion could, in the presence of oxygen, lead to formation of nickel molybdate, and diffusion of the nickel into the molybdenum could result in recrystallization of a surface layer in the molybdenum-rich alloy.

Section 8. 'Self-Healing' Characteristics of Aluminium-Chromium-Silicon Coating

An attempt was made to define, quantitatively, the reported 'self-healing' properties of defective aluminium-chromium-silicon coatings under oxidizing conditions.

Section 9. The Nature of Promising Coatings

Throughout the investigation consideration was given to the mechanism by which the more promising coatings exercise their protective effect. In this section the test data are reviewed, in an attempt to define the nature of the protection afforded by (*a*) aluminium-bonded coatings (exemplified by the aluminium - chromium - silicon composition), (*b*) nickel-base alloy coatings (represented by nickel-chromium-boron and nickel-silicon types), and (*c*) electrodeposited coatings of chromium and chromium + nickel.

Conclusions

The main conclusions drawn are summarized below.

The ballistic-impact resistance of electrodeposited chromium coatings was shown to be satisfactory.

Electrodeposited chromium coatings, aluminium-chromium - silicon, nickel - chromium - boron, and nickel-silicon-boron coatings, and a composite of nickel-silicon-boron + aluminium-chromium-silicon withstood 2 per cent. elongation at 1800°F. (980°C.) without failure.

In an erosion test at 1800°F. (980°C.) all the coatings mentioned in the previous paragraph, and an aluminium-nickel-silicon coating, withstood the erosive action of hot grit for at least 25 minutes.

Thermal-shock tests of composite and modified coatings gave encouraging results for porous chromium electrodeposits having a superimposed spray coating of aluminium-chromium-silicon. Such composites are considered to warrant further investigation.

In all the tests except those involving thermal cycling, electrodeposited chromium/nickel coatings on molybdenum proved equal or superior to other coatings tested. The thermal-cycling test revealed a specific limitation to this type of coating, because the nickel separated from the underlying chromium.

Nickel cladding showed satisfactory resistance to ballistic impact, but was less resistant to oxidation than nickel-base coatings containing chromium and silicon. Improvements in edge protection are necessary before the effectiveness of the cladding can be fully evaluated.

Room-temperature ductility of molybdenum, in bend tests and in special tup-type impact tests, was lowered by coating with aluminium-chromium-silicon and with nickel-chromium-boron alloys. The ductility of the base 0·5 per cent. titanium molybdenum alloy was unaffected by exposure for 500 hours at 1800°F. (980°C.) + 25 hours at 2000°F. (1095°C.).

Oxidation tests at temperatures up to 3000°F. (1650°C.) established the following tentative limits for promising sprayed metal coatings on molybdenum.

Aluminium-chromium-silicon	2600°F. (1426°C.)
Nickel-chromium-boron	2200°F. (1205°C.)
Nickel-silicon-boron	2200°F. (1205°C.)

Coatings comprising composites or mixtures of aluminium and aluminium-oxide showed no advantage over the metallic coatings at high temperatures of test.

Use of a diffusion barrier layer of copper between the molybdenum base and nickel-alloy coatings was unsuccessful.

Study of the 'self-healing' of aluminium-chromium-silicon coatings indicates that, under some test conditions, the flow of a eutectic phase from the coatings can re-establish a cover at a position where mechanical damage has exposed small areas of molybdenum.

The following summary is made of the behaviour and nature of the general types of coating studied.

(*a*) *Aluminium-bonded coatings.* These coatings are characterized by excellent bonding to the molybdenum base through formation of a Mo_3Al-type layer which delays oxidation of the underlying molybdenum. In evaluation tests at 1800°F. (980°C.), the coatings exhibited good resistance to oxidation and thermal shock, moderate resistance to erosion, but poor resistance to ballistic impact.

(*b*) *Nickel-base coatings.* These are ductile coatings which serve well until cracks occur, after which oxidation, through cracks, results in formation of

nickel molybdate, which accelerates failure under thermal-cycling conditions. In evaluation tests at 1800°F. (980°C.) the coatings exhibited good resistance to oxidation and ballistic impact.

(*c*) *Chromium and Chromium/Nickel Electrodeposited Coatings.* Cracks in the chromium promote pit-type attack on the underlying molybdenum: the presence of a nickel overlayer delays this attack. In tests at 1800°F. (980°C.), these coatings exhibited good resistance to ballistic impact and erosion, erratic behaviour in oxidation tests, and poor resistance to thermal shock.

Corrosion by Vanadium Pentoxide: Influence of Titanium in High-Alloy Materials

A. BURDESE and S. GALLO: 'Corrosion of Metals at High Temperatures by Vanadium Anhydride.'

Metallurgia Italiana, 1958, vol. 50, Jan., pp. 8–14.

Following observations of the protective influence of titanium in improving the corrosion-resistance of certain types of steel, tests were carried out to ascertain the effect of titanium content on resistance, to vanadium-pentoxide attack, of chromium, cobalt or nickel alloys, and of high-alloy chromium-nickel steels. Titanium ranged, in most of the series, from trace amounts up to 8–10 per cent. The materials were subjected to tests of 300-hours' duration, at 700°C., in an atmosphere of V_2O_5. The degree of attack was studied as a function of the titanium content of the alloys, and the nature of the corrosion undergone was further investigated by examination of the structural changes accompanying corrosion.

It is concluded that titanium has a progressively beneficial effect, due to the comparative stability of the protective film formed on the alloys in which substantial amounts of the element are present. The effects can be better utilized in the high-chromium or high-cobalt alloys than in the high-nickel types, since in the nickel-rich compositions the presence of substantial amounts of titanium induces brittleness.

Tau- and Y-Phases in Austenitic Steels

O. KNOP: 'The Identity of the Tau- and Y-Phases from Austenitic Steels.'

Metallurgia, 1958, vol. 57, Mar., pp. 137–8.

BROWN, CLARK and PARKER recently described a phase isolated from chromium-nickel-titanium steels by electrolytic extraction (*ibid.*, 1957, vol. 56, Nov., pp. 215–23). The phase, designated *tau*, was considered to be identical with the *Y*-phase reported by GEMMILL *et al.* (*Jnl. Iron and Steel Inst.*, 1956, vol. 184, Oct., pp. 122–44). In this correspondence the writer details some of the factors which lead him to believe that such an assumption is not warranted on the basis of the evidence presented.

In reply, BROWN, CLARK and PARKER confirm their previous findings that comparison of the diffraction patterns of *tau*-phase and of *Y*-phase leaves little doubt that the two are virtually identical.

Effect of 'Sensitizing' Treatment on Mechanical and Physical Properties of Stainless-Steel Wire

S. STORCHHEIM: 'Effect of 475°C. Heat-Treatment on Cold-Worked 18-8 Stainless-Steel Wire.'

Wire and Wire Products, 1958, vol. 33, Feb., pp. 172–3, 222.

The phenomenon of impaired resistance to intercrystalline corrosion resulting from exposure in the sensitizing temperature range (400°–900°C.) is well known. This paper deals with the influence of such thermal treatment on properties other than corrosion-resistance, viz., specific electrical resistance, tensile strength, magnetic remanence, and coercive force. The study was made on '18-8'-type chromium-nickel steel wire, which had been cold-worked from 0·028 to 0·004 in. (0·7 to 0·1 mm.) diameter, i.e., a 98 per cent. reduction. The carbon content of the steels varied from 0·063 to 0·120 per cent. The wires were strand heat-treated by passing through a furnace in conditions which involved maintaining at temperature (about 475°C.) for only 2 seconds. The author draws attention to the fact that precipitation occurred in that short period, although precipitation treatments as such are normally of much longer duration.

The results are shown in a series of graphs and tables which indicate the influence of carbon content on the behaviour of the wire under the stress-relief/precipitation treatment. The following are among the main conclusions drawn:

Electrical Resistance

With increasing carbon there is a fall in electrical resistance, but at about 0·07–0·08 per cent. of carbon the curve levels considerably. With low carbon the 475°C treatment caused increase in resistance, but with increasing carbon resistance eventually decreases. The initial increase may be attributed to stress-relief, while the decrease is caused through precipitation of carbide.

Tensile Strength

With low carbon the influence of the heat-treatment is negative, due to the predominating effect of stress-relief, but as the carbon rises the effect of such heating is to increase the tensile strength, as a result of precipitation effects.

Magnetic Remanence

This property was the most drastically affected of the characteristics studied. With rise in carbon content B_r fell very rapidly, but the rate of fall decreased at about 0·07 per cent. carbon.

Coercive Force

Since the data on this property are not considered to be so authoritative as those relating to the other changes, no plot was made of the information obtained. It is, however, noted that there was some indication that with increasing carbon content, the thermal treatment applied causes decrease in coercive force.

The changes resulting from the 475°C. treatment were of considerable magnitude, e.g., in specific

resistance 10 per cent., in ultimate tensile strength almost 8 per cent., and for magnetic remanence 40 per cent. The changes indicate that, for commercial applications, it is important to study the effect which stress-relief and possible precipitation of carbides can have upon physical properties.

It is also of interest to note that for the properties in which both negative and positive effects were produced by such treatment, the change of sign occurs in all cases at about 0·06–0·07 per cent. carbon. Below this level the stress-relief phenomenon is the governing one, whereas with higher carbon the effect of carbide-precipitation predominates.

Resistance of Nickel-containing Materials to Corrosion by Amine-Gas-Treating Solutions

F. S. LANG and J. F. MASON: 'Corrosion in Amine-Gas-Treating Solutions.'

Corrosion, 1958, vol. 14, Feb., pp. 105t–8t.

Various ethanolamines, such as the mono-, di- and tri-ethanolamines, have general chemical properties closely analogous to those of ammonia, and when used in the pure state or in solutions with water or glycol, at moderate temperatures, they are not actively corrosive to cast iron and mild steel. Corrosion problems have, however, been introduced by the development of the amine process for the removal of acid gases (e.g., hydrogen sulphide and carbon dioxide) from natural and refinery gases, and by the amine-glycol process for the removal of gases and water from petroleum products.

This paper reports results of some laboratory experiments designed to ascertain the fundamental causes of corrosion problems arising in amine-gas-treating systems. These experimental findings are supplemented by information obtained from corrosion tests made in operating-plant conditions, and by information relating to the use of amines in stripping acid gases from hydrocarbon streams. The laboratory work covered corrosion tests on a number of materials in decomposition products of monoethanolamine, study of the effect of the presence of iron-sulphide sludge in the amine, and investigation of the damage caused by high-velocity flow and turbulence.

In most of the tests a comparison was made of the behaviour of the following materials: 'Monel', nickel, 'Inconel', 'Incoloy', austenitic nickel-chromium-(molybdenum) steels, high-chromium steel, and mild steel. In some of the plant exposures aluminium, 'Ni-Resist' and plain cast iron were also included.

The results, reported in terms of inches penetration per year, with notes on the nature of the corrosion undergone, lead to the following conclusions:

In the absence of acid gases, such as carbon dioxide and hydrogen sulphide, ethanolamine solutions can be safely handled in plain cast-iron and steel equipment. In amine solutions saturated with those gases corrosion can be expected to increase with amine concentration, temperature, and pressure.

Contactors or absorbers of mild steel are relatively free of corrosion in amine-treating service, but should be stress-relieved before being put into service. In re-boilers, re-activators, heat exchangers, pumps, and related piping the corrosion rate of steel may be excessive.

In laboratory tests 'Monel' was shown to be satisfactory in amines saturated with carbon dioxide or hydrogen sulphide alone, but could be corroded by gas mixtures of approximately equal proportions. In clean solutions, at relatively low velocities, Types 304 and 316 stainless steel were substantially uncorroded.

Under plant conditions, complicating factors such as turbulence, high velocity, sludge, hot-wall effects, and decomposition products of the amine can increase the corrosion attack. Nickel-chromium stainless-steel tubes have been observed to fail by erosion, corrosion or impingement attack. Iron-sulphide sludge has been found to be more noble than 'Monel' and stainless steel, and to increase the corrosion of these materials.

By improvement of plant practice (e.g., filtering-out sludge and re-distilling part of the amine), avoidance of unsuitable temperatures, pressures, concentrations and velocities, and correct selection of materials of construction (e.g., 'Monel' or stainless steel) good resistance to corrosion can be secured in the types of plant considered.

Resistance of Stainless Steel to Stress Corrosion by Geothermal Steam

T. MARSHALL: 'Stress Corrosion of Austenitic Stainless Steel in Geothermal Steam.'

Corrosion, 1958, vol. 14, Mar., pp. 159t–62t.

Commercial exploitation of geothermal steam for use in production of electrical power has in recent years been actively pursued by the New Zealand Government. In this connexion, it has been necessary to establish the probable behaviour of metallic materials under the conditions which would be involved in handling the steam: the results of preliminary tests, made on a wide range of materials, were reported by the present author and HUGILL in *Corrosion*, 1957, vol. 13, pp. 329t–37t (*Nickel Bulletin*, 1957, vol. 30, No. 7–8, p. 142).

Wet steam, typical of that found in the Wairakei district, was used in the tests, and, since this steam is severely contaminated with chlorides, hydrogen sulphide, carbon dioxide, etc., it was considered that stress-corrosion cracking was one of the problems which might be anticipated in using austenitic nickel-chromium steels in such conditions. Only two cases of such cracking have, however, occurred, and these were only as a result of exposure to geothermal steam which had been deliberately contaminated with oxygen by injection of air. The question then arose as to whether these results could be regarded as experimental corroboration of the postulation of HOAR and HINES that cathodic inhibition by complete exclusion of oxygen would prevent transcrystalline stress corrosion of austenitic steels. (See *Jnl. Iron and Steel Inst.*, 1956, vol. 182, Feb.,

pp. 124–43; *Nickel Bulletin*, 1956, vol. 29, No. 5, pp. 96–7.) Further tests, the subject of this paper, were therefore carried out with the specific aim of studying more closely the effect of oxygen contamination on stress corrosion of austenitic stainless steels in geothermal steam. The work is considered of considerable practical importance, since previous investigations have emphasized the advantages of the austenitic stainless steels *vis-à-vis* the martensitic/ferritic grades. The tests were made on austenitic chromium – nickel, chromium – nickel – molybdenum and chromium-nickel-niobium stainless steels of '18-8' type; 13 per cent. chromium and chromium-molybdenum stainless steels; and (for purposes of comparison) 'Monel', 'Inconel', 60–40 brass, titanium, aluminium and mild steel. Specimens were of the U-bend type and the tests were made at 150°C., i.e., at a nominal superheat of 25°C., in (1) air-free geothermal steam, and (2) aerated steam. Full details are given of test conditions and procedure and of the data obtained.

The investigation establishes clearly that austenitic stainless steels, even under severe applied stresses, are resistant to stress-corrosion cracking in air-free geothermal steam heavily contaminated with chlorides and hydrogen sulphide. Since, however, stress corrosion occurs readily when the steam is moderately superheated and simultaneously contaminated with oxygen by aeration, the results are regarded as confirmatory of the theory of Hoar and Hines. Further corroboration was obtained by tests on austenitic steels in air-free steam varying from 0°C. to 31°C. in which no stress-corrosion cracking occurred.

From consideration of the present results and of those previously published by other investigators, the author concludes that the simultaneous existence of five conditions is needed to cause stress-corrosion cracking to occur in geothermal steam. The conditions are:

(1) The steel must be in a state of tensile stress.

(2) A water phase must be present in the steam. (In the absence of a high degree of superheating this phase would normally be present.)

(3) The steel must be exposed at an elevated temperature. (The minimum temperature necessary for cracking has been quoted as 50°C., and at pressures in excess of 2 p.s.i.a.* this temperature would necessarily be present.)

(4) The water phase of the steam must contain chlorides.

(5) Oxygen must be present in the steam.

It is emphasized that all five conditions must exist simultaneously to cause stress-corrosion cracking in the austenitic nickel-chromium steels, and that in a turbine operating on geothermal steam such an occurrence would be extremely rare. It is therefore considered that these materials are particularly promising for turbine blading in this application: in this connexion the hydrogen-sulphide content of the steam, with its accompanying reducing action, is an important factor.

It is emphasized that the conclusions drawn with regard to the conditions essential for onset of stress-corrosion cracking may be applied with confidence only to the geothermal steam used in the tests, although further investigations indicate that they might be applicable also to other steam systems, such as boiler-steam plants.

p.s.i.a. = lbs. per sq. in. (absolute), in distinction to *p.s.i.g.*, which signifies lbs. per sq. in. (gauge).

Corrosion of Metals in a Marine Tropical Atmosphere

B. W. FORGESON, C. R. SOUTHWELL, A. L. ALEXANDER, H. W. MUNDT and L. J. THOMPSON: 'Corrosion of Metals in Tropical Environments.'
Corrosion, 1958, vol. 14, Feb., pp. 73t–81t.

Paper presented to Nat. Assocn. Corrosion Engineers, Mar. 1957. The subject matter is identical with that reported in *U.S. Naval Research Laboratory, Washington, Report N.R.L.* 4929, June 19, 1957; see abstract in *Nickel Bulletin*, 1958, vol. 31, No. 2, p. 59.

Corrosion of Metals in Industrial Atmospheres

G. SCHIKORR: 'The Corrosion of Metallic Materials and Protective Coatings in Industrial Atmospheres.'
Schweizer Archiv, 1958, vol. 24, Feb., pp. 33–46.

The paper comprises a review of information on the corrosion of metals under exposure to industrial atmospheres. Reference is made to experience in many parts of the world, and the survey is supported by a *bibliography* of 78 items.

The review falls into three sections. The first comprises a general survey of the influence, on rate of corrosion, of variables such as the composition of the air, the gaseous and solid impurities present, humidity, etc. The information available is critically discussed, in an attempt to trace some relationships among the multitude of phenomena which have been reported. The second section centres on the behaviour of individual non-ferrous metals, and their suitability for use as protective coatings for less resistant materials: in this connexion reference is made, *inter alia*, to nickel and electrodeposited nickel coatings. The final section deals with the corrosion of iron and its alloys and the effect, on rate of corrosion, of the formation of layers of rust. Mention is made of the excellent resistance of the 18–8-type chromium-nickel stainless steels to corrosion by industrial atmospheres.

Corrosion-Resisting Chromium-Nickel-Manganese Steel

AMER. SOC. TESTING MATERIALS: 'Tentative Specifications for Corrosion-Resisting Chromium-Nickel-Manganese Steel Plate, Sheet and Strip.'
A.S.T.M. Designation A. 412–57*T*.
Supplement to *Book of A.S.T.M. Standards, Pt. I, Ferrous Metals*, 1957, pp. 192–202.

The specifications cover soft and high-strength plate, sheet, and strip of two compositional types

A.S.T.M. Specifications for Chromium-Nickel-Manganese Stainless Steel

(*See abstract on p. 147*)

Type	C %	Si %	S %	P %	Cr %	Ni %	Mn %	N %
201	0·15 max.	1·00 max.	0·030 max.	0·060 max.	16·00-18·00	3·50-5·50	5·50-7·50	0·25 max.
202	0·15 max.	1·00 max.	0·030 max.	0·060 max.	17·00-19·00	4·00-6·00	7·50-10·00	0·25 max.

(A.I.S.I. Types 201 and 202). Type 201 is intended for use where ability to undergo severe forming operations is not of major importance: Type 202 is for applications in which such characteristics are of primary significance. The composition elements laid down for the two grades are given above.

Tensile requirements are specified, as are bend tests, dimensional and other tolerances, and the various types of finish in which the materials may be supplied.

Corrosion by Fuming Nitric Acid: Effect of Inhibitors

T. B. YEE: 'Effect of Inhibitors in Fuming Nitric Acid on Corrosion and Oxidation.'

Corrosion, 1958, vol. 14, Feb., pp. 82t-4t.

Addition of inhibitors to fuming nitric acid has been found, in some cases, to affect the oxidizing power of the acid. This paper reports a systematic study of the influence, on corrosive quality and oxidizing power, of a variety of inhibiting media. The test material exposed to the inhibited-acid media was a niobium-stabilized nickel-chromium stainless steel. The inhibitors studied were KI, KIO_3, I_2 I_2O_5, CaF_2, HF, NH_4F, NaF, and Na_3AlF_6. The following inhibitors (0·5 per cent.) were found to increase the oxidizing power of the acid: I_2, I_2O_5, KI, CaF_2, NH_4F and HF. The reverse effect was obtained by addition of 1 per cent. KI, 0·5 per cent. KIO_3, 5 per cent. HF, 1 per cent. HF, 0·5 per cent. NaF and 0·5 per cent. Na_3AlF_6. It is concluded that the most satisfactory corrosion-inhibiting media are KI, KIO_3, I_2 and I_2O_5

Effect of Irradiation on Resistance of Aluminium-Nickel Alloy to Corrosion by High-Temperature Water

E. L. MARTINEC: 'Corrosion of an Aluminium-Nickel Alloy in a Reactor Test Loop.'

U.S. Atomic Energy Commission Report ANL-5783, Sept. 1957; 18 pp.

Initial studies by Draley and Ruther indicated that addition of small amounts of nickel to aluminium conferred local cathodic protection at grain boundaries and that, in particular, an aluminium-nickel alloy containing 1 per cent. nickel showed considerably improved resistance to corrosion by high-temperature water (see *Nickel Bulletin*, 1956, vol. 29, No. 7, p. 145). Little work had been carried out on the effect of irradiation on the corrosion-resistance of such alloys and tests which had been made were too short to prove conclusive. The investigation described in this report was therefore planned to determine the resistance, to corrosion by high-temperature water, of an aluminium-base alloy containing 1 per cent. nickel, under conditions of long-term irradiation similar to those encountered during operation of a nuclear reactor. The project formed part of a series of investigations being carried out, at the Argonne National Laboratory, into the use of aluminium alloys as materials of construction for nuclear power plant.

Tests were carried out in a specially constructed high-pressure water loop; the temperature of the water was maintained within the range 470°-485°F. (245°-250°C.) during irradiation.

The results showed that weight loss per unit time is the same for both the in-pile irradiated specimens and the out-of-pile control specimens which were not exposed to irradiation. The control specimens, however, were found to have a higher initial corrosion rate. It is urged that the usefulness of the 1 per cent. nickel-aluminium alloy would be enhanced by modification to combine a higher degree of mechanical strength with its proved corrosion-resisting properties.

Fatigue Properties of Precipitation-Hardening Stainless Steels

M. M. WEISMAN, J. MELILL and T. MATSUDA: 'Uni-Directional Axial-Tension Fatigue Tests of Beryllium-Copper and Several Precipitation-Hardening Corrosion-Resistant Steels.'

Contained in *Symposium on Fatigue of Aircraft Structures*, published as *A.S.T.M. Special Tech. Publn. No.* 203, 1957, pp. 47-60; disc., pp. 61-6.

For many years the low-alloy steels and high-strength aluminium alloys have been the conventional constructional materials for aircraft, but the higher performance being called for in modern units is necessitating the introduction of materials of higher strength, characterized, in some cases, by properties of a specialized nature.

Information on the properties of such materials is, at present, inadequate to permit of drawing up specifications and there is urgent need for compilation of experimental test results which will provide a sound basis for design. The information given in this paper forms a contribution towards assembly of the necessary data. The results of notched tensile tests and of smooth and V-notched fatigue

tests are reported for beryllium-copper, 'AM-350' chromium-nickel-molybdenum steel, and 'A-286' high-alloy nickel-chromium steel. Tests on open-hole notched specimens of '17-7 P.H.' nickel-chromium steel are also included. Behaviour of single-lap riveted aircraft joints is simulated by cyclic-loading tests on dimpled and riveted sheet specimens of the 'AM-350' steel, and tests are reported on '17-7 P.H.' specimens containing unloaded rivet-filled holes.

The results of the tests are discussed from the standpoint of an engineer-designer in the aircraft industry.

Stainless-Steel and Titanium Sandwich Structures

W. J. LEWIS, G. E. FAULKNER and P. J. RIEPPEL: 'Stainless-Steel and Titanium Sandwich Structures.' *U.S. Atomic Energy Commission, Battelle Memorial Institute Report TML No.* 79, Aug. 9, 1957; 34 pp. *P.B.* 121633.

The need for materials having high strength-to-weight ratios (particularly types which would be light enough to be suitable for aircraft structures and yet strong enough to withstand exposure to the elevated temperatures involved in high-speed flight) has concentrated efforts on the development of stainless-steel and titanium 'sandwich' structures.

The aim of this survey was 'to collect and disseminate information of a general nature concerning (1) the fabrication of stainless-steel and titanium sandwich panels, (2) the problems encountered in fabricating panels, and (3) the development work under way'. The work was the result of a literature search of published U.S. Government and industrial reports, and of interviews with many people in companies engaged in the fabrication or development of such structures.

The information on stainless steel relates mainly to '17-7 P.H.' Consideration is, however, also given to the possible use of such steels as 'A-286', 'AM-350' and the A.I.S.I. Types 310 and 321 chromium-nickel austenitic steels. The review comprises two parts. The first outlines procedures used to fabricate the three basic types of sandwich structure: honeycomb-core, corrugated-core, and waffle-core. In this context reference is made to fabrication of the cores, edge members and panels, and to qualification and inspection requirements. The second section, covering development work currently in progress, relates mainly to improvements in brazing, welding and adhesive bonding.

Little information is available on titanium sandwich structures, since none is yet in production. A brief résumé is, however, given of the efforts being made to overcome the problems which are inhibiting progress: these include the non-availability of titanium-alloy foil and lack of a suitable process for fabricating the panels.

Classification of Filler Metals Conforming to A.W.S./A.S.T.M. Specifications

See abstract on p. 124.

'Curtain-Wall' Construction: Use of Stainless Steel

'Curtain Wall Do's and Dont's.'

Report of Proceedings at Seminar held January 29, 1957, at the Engineering Societies Building, New York.

This informal seminar was planned to bring together those interested in curtain-wall construction, taking into consideration both the design and the materials aspects.

A curtain wall is defined as 'a non-load-bearing wall consisting of three elements: (1) a window, either fixed or operating; (2) a panel, either spandrel or mullion; and (3) a frame unit which ties the window and panel to the building.' Within the scope of this definition, contributors to the discussion presented papers dealing with materials which can be used in such construction, and with fabrication and erection of curtain walls, design, etc. Materials considered included aluminium and its alloys, chromium-nickel stainless steel, and glass, and the discussion also included questions of insulation and sealing, and the media used for such purposes.

The paper relating to Stainless Steel was presented by R. PARET, who opened by drawing attention to the outstanding advantages of such materials for architectural uses: first, their strength and, second, their durability. After emphasizing the easy availability and fabricating qualities of the austenitic steels, some recommendations were made with regard to design criteria which should be adopted for such materials, with particular reference to control of optical distortion (e.g., in relatively thin sheet by elimination or minimizing of flat areas, and by use of curved vertical panels which absorb dimensional changes due to fluctuations of temperature). The author cited examples of buildings in which successful methods have been adopted. If complete visual flatness is required, heavier material, stiffened panels, or backed steel may be used.

The importance of surface finish has been recognized: a wide variety of finishes suitable for architectural use can now be obtained, and roll-forming and texturing is being employed to obtain contrasting effects. In connexion with colour, attention was drawn to oxidation treatments which can be applied to stainless steel and to the attractive effects which can be obtained by selective enamelling.

'Monel' Equipment for Handling Fluorine

Industrial and Engineering Chemistry for February 1958 includes a series of papers dealing with nuclear technology in the chemical and petroleum industries. The three items referred to below have particular interest in relation to nickel-containing materials.

S. P. VAVALIDES, R. E. CABLE, W. K. HENDERSON and C. A. POWELL: 'High-Capacity, Long-Life Fluorine Cell', pp. 178-80.

In view of the rapidly increasing demand for fluorine, particularly in connexion with the atomic energy programme, considerable effort is being directed

towards development of more efficient fluorine-manufacturing units. This paper describes the construction and operating details of a new 32-anode fluorine cell which operates at 4,000–6,000 amperes. The following details are given of the materials used for the respective components:

Materials used in 89 × 32 × 39-*inch Cell*

Components	*Material*
Shell	'Monel' liner, steel jacket
Head	Steel plate, 'Monel' skirt
Anodes	Carbon
Cathode	Steel
Diaphragm	'Monel'
Anode support bar	Chrome-molybdenum steel (A.I.S.I. 4140)
Anode cap screws	Chrome-molybdenum steel (A.I.S.I. 4140)
Anode pressure plates	Copper

Some account is given of problems encountered in operation of the cell, the majority of which have been due to corrosion of steel and copper components in the anode-joint assembly, which resulted in loss of electrical contact between the carbon/metal joint and subsequent operation of the cell at higher voltage. Development work is being carried out to ensure the use of materials which will give better resistance to corrosion by the potassium-bifluoride/hydrogen-fluoride electrolyte.

J. DYKSTRA, B. H. THOMPSON and W. C. PARIS: 'A 25-Pound-per-Hour Fluorine Plant', pp. 181–6.

The authors describe plant and process used for production of fluorine at the Oak Ridge Gaseous Diffusion Unit. The cell is of a medium-temperature type, using a fused-salt electrolyte (KF.2HF), carbon anodes and steel cathodes. In this cell also the shell is a steel jacket with 'Monel' liner, and the diaphragm is also of 'Monel'. The cell, which is fully described and well illustrated, is a modified version of the Union Carbide Chemical Company's cell (*ibid.*, 1955, vol. 47, pp. 878 and 883), having increased cooling capacity, greater effective anode-cathode area and an improved system for collection of effluent gases. An important feature is the piston-type compressor, the advantages of which are discussed.

R. P. MILFORD: 'Engineering Design of Oak Ridge Fluoride Volatility Pilot Plant', pp. 187–91.

The paper opens with a review of the essential stages in the fluoride volatility process, which is considered promising as a means of recovering uranium from nuclear-reactor fuel elements of the zirconium type. The reactions involved in the process are detailed, and a flow sheet shows its various stages. A description is then given of a pilot plant at Oak Ridge National Laboratory, which will recover uranium from spent zirconium-enriched uranium reactor fuels by volatilization of uranium hexafluoride and absorption on sodium fluoride. Reference is made, *inter alia*, to the selection of extra-low-carbon nickel ('L' Nickel) for the fluorinator, use of nickel piping for passage of fluorine into the vessel, and choice of 'Monel' as resistant to corrosion in conditions involved in handling uranium hexafluoride.

Welded Stainless-Steel Tanks for Plastics-Manufacturing Plant

'Welding of Twelve Stainless-Steel Tanks for I.C.I.'
Chemical Age, 1958, vol. 79, Mar. 15, p. 502.

This note describes tanks supplied to the Plastics Division of Imperial Chemical Industries, Ltd., for storage of an unspecified 'corrosive liquid'.

Two tanks used earlier for this purpose were made from mild steel lined with rubber, but this lining was found not to give the required protection against corrosive attack by the liquid stored. The main barrels of the remaining ten tanks were therefore fabricated from 18–8 type chromium-nickel stainless steel. In order to avoid scrapping the two mild-steel tanks, both were lined with 18–8 stainless steel. This procedure presented some difficulties, due to the fact that the tanks had already been used, and on account of the sizes of commercially available lining sheets. The methods adopted for lining are described. All twelve tanks are reported to be giving satisfactory service.

Glossary of Terms used in Reference to Corrosion

'Inter Society Corrosion Committee Glossary of Corrosion Terms.'
Corrosion, 1958, vol. 14, Feb., pp. 71t–2t.
N.A.C.E. Publn. 58–5.

This glossary, submitted by the N.A.C.E. Sub-committee on Standard Definitions and Terminology, at the 1957 meeting of the Society, contains proposed definitions of 53 terms commonly used in connexion with corrosion. Of these terms, 18 are taken from the *Corrosion Handbook* published by *John Wylie & Sons, New York*, 1948; the remainder are original proposals by the sponsoring committee.

Determination of the Acid-Resistance of Steels by the Potentiostat Technique

C. EDELEANU: 'A Potentiostat Technique for Studying the Acid-Resistance of Alloy Steels.'
Jnl. Iron and Steel Inst., 1958, vol. 188, Feb., pp. 122–32.

The author has earlier given details of a potentiostat technique which may be used for evaluation of the corrosion-resisting characteristics of steel (*Metallurgia*, 1954, vol. 50, pp. 113–16; *Jnl. Iron and Steel Inst.*, 1957, vol. 185, pp. 482–8). In the present paper this method of assessment is applied to the study of several types of corrosion-resisting steel, exposed under various conditions of attack. It is pointed out that the corrosion-resistance of metallic materials cannot be classified on any absolute basis, but that characteristics and properties vary (often to the extent of giving 'good' or 'bad' behaviour, for any one material) according to the conditions of

exposure (corroding medium, temperature, aeration, duration of exposure, etc.).

The general term 'acid-resistance' must be considered as a multiple generic, the significance of which is contingent on several variants. The potentiostat technique has been devised to study such contributory factors, and thereby to provide a basis which will permit of assessment of behaviour under given conditions. The properties of typical high-alloy steels are considered under the following headings:

Resistance to highly oxidizing solutions
Range of passivity
Rate of corrosion of the passive metal
Stability of passivity
Maximum rate of corrosion
Relative nobility

The essential features of the potentiostat technique are described, with illustrations of the apparatus used, and its application is discussed on the basis of experiments on some experimental steels. Individual tests in the series considered involved exposure under highly oxidizing conditions, to non-oxidizing acids containing air, and to acids under reducing conditions.

Cupric-Chloride Modification of the Acetic-Acid Salt-Spray Test

See abstract on p. 131.

Measurement of Corrosion by Gaseous Media: Nickel/Chlorine Reactions

See abstract on p. 127.

Direct-Reading Corrosion Meter

G. A. MARSH and E. SCHASCHL: 'Some Considerations in the Design and Application of an Electrical-Resistance Corrosion Meter.'

Corrosion, 1958, vol. 14, Mar., pp. 155t–8t.

After summarizing, with due reference to the literature, previous work which has been carried out in this field, the authors describe the development and main features of a corrosion meter by means of which loss in thickness of a corroding metal can be instantly determined by measurement of electrical resistance. The specimens or probes are connected as two arms of a Wheatstone bridge, one arm being protected by a suitable coating, while the other is exposed to the corrodent. (Since both arms are of the same metal, the influence of temperature on the electrical-resistance values is cancelled out.) The loss in thickness is indicated directly on a dial, in penetration units.

The main advantages claimed, by comparison with conventional corrosion-test methods, are improved sensitivity and reproducibility, the fact that measurements can be made without interruption to the test, and the possibility of carrying out a number of tests with the one apparatus. The method cannot, however, be used to measure pitting corrosion.

The paper also reviews laboratory and field applications for which this test technique is suitable, special emphasis being placed on those which are of significance for the petroleum industry.

'Fescolizing' of Stabilizer Parts

See abstract on p. 131.

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Registered Trade Marks*.

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